# JOURNAL OF DAIRY SCIENCE

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Vol. XXIII, No. 3, March, 1940

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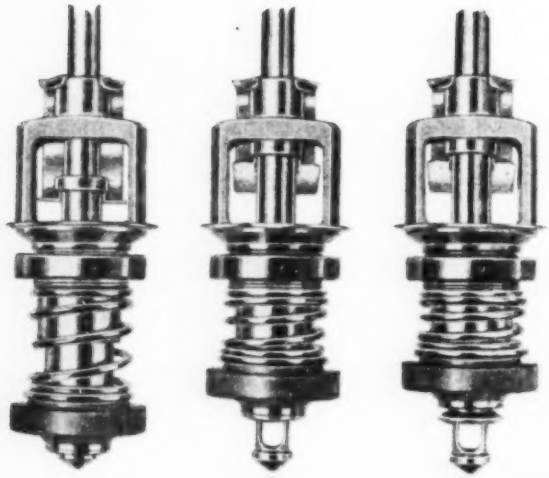


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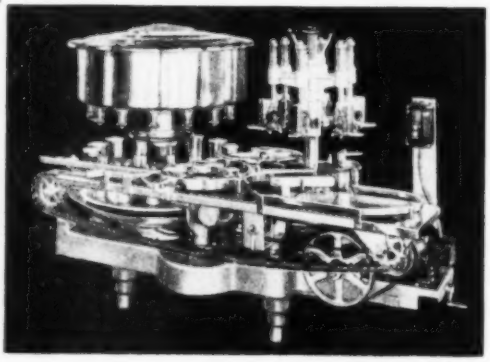

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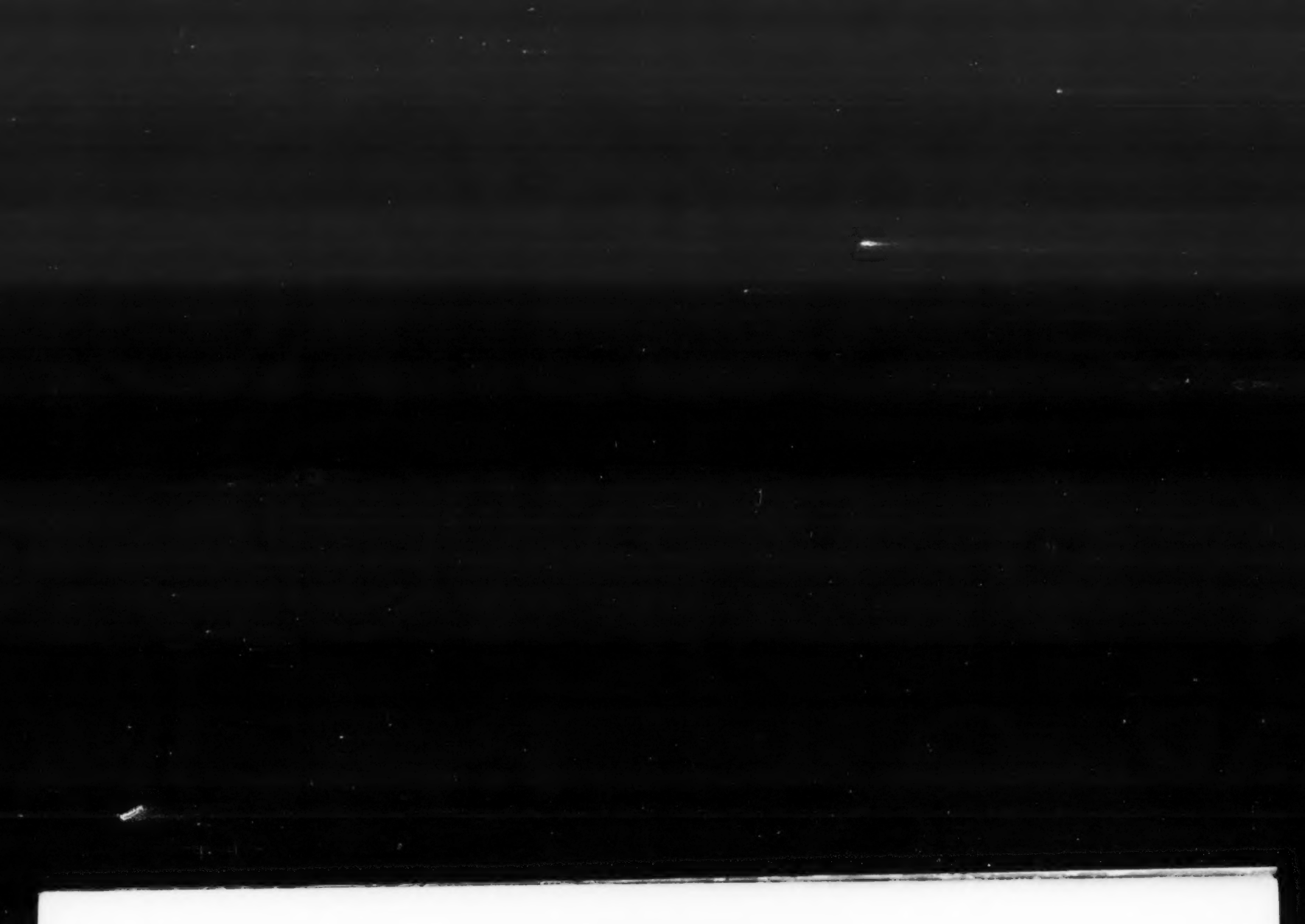

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*The Journal of Dairy Science* is issued monthly. Subscription is by the volume and one volume is issued per year.

*Manuscripts* should be typewritten and carefully revised before submission to T. S. Sutton, The Ohio State University, Columbus, Ohio. Twenty-five reprints will be furnished gratis to authors. Cost of additional reprints and reprint order blank will be submitted with proof.

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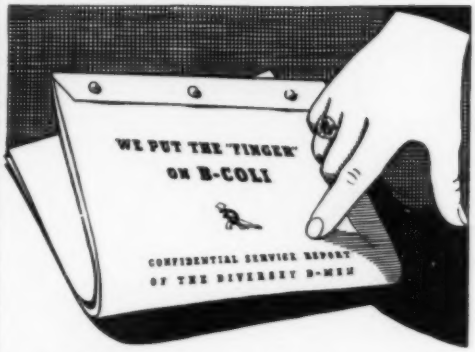


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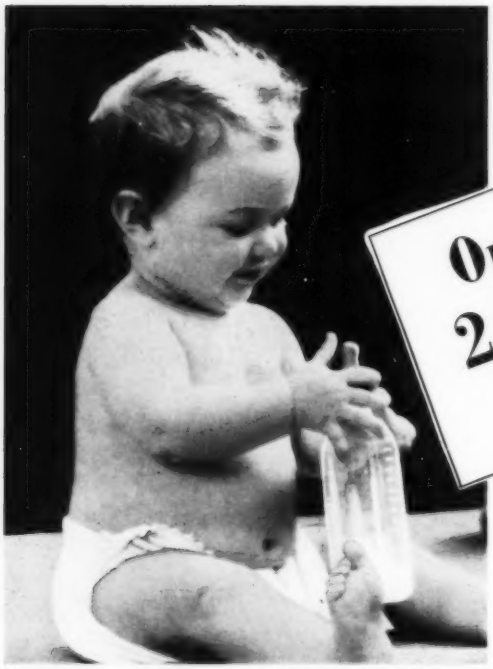


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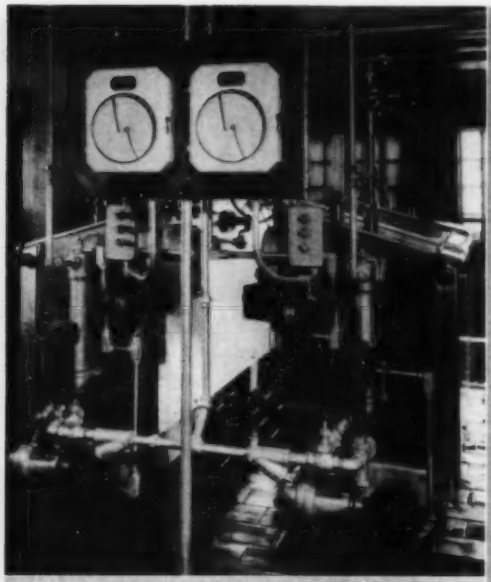

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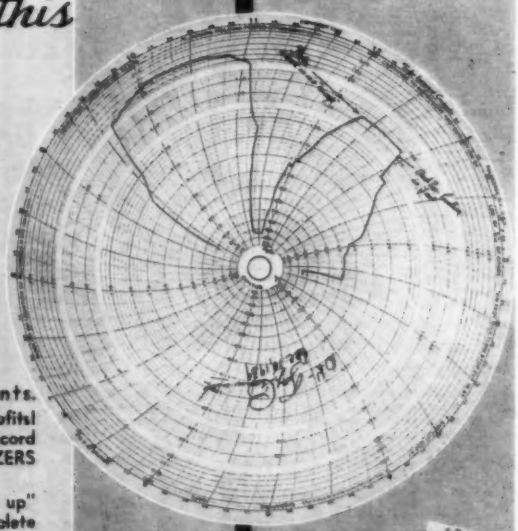

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JOURNAL OF DAIRY SCIENCE

VOLUME XXIII MARCH, 1940 NUMBER 3

# DETERMINATION OF FAT, MOISTURE, AND SALT IN SOFT CHEESE

G. H. WILSTER, CHAIRMAN, W. V. PRICE, A. J. MORRIS, E. F. GOSS, G. P. SANDERS

*The Subcommittee for the Analysis of Cheese, A. D. S. A.*

Methods for determining the fat, moisture and salt contents of hard cheese were reported by the committee in 1936. They were published in Volume XX, No. 1, of the JOURNAL OF DAIRY SCIENCE.

The methods outlined below are applicable in analyses of such varieties as Limburger, Brick, Roquefort, Bel Paese, cottage, Neufchatel, cream, Camembert, etc., or such varieties as are characteristically sticky. In analyzing samples of cheese which are more firm in texture, the methods previously outlined for hard cheeses (1) are used. All analyses should be made in duplicate.

1. *Sampling:*

When the cheese can be cut, take a narrow wedge-shaped segment reaching from the outer edge to the center. If the loaf or block is small, cut it through the center and remove a slice of suitable thickness to provide a sufficient amount of sample, from the freshly cut surface. If the cheese, or the contents of a package, is too small for obtaining a slice the whole cheese or the total contents of the package must be used. Cut the sample into strips and pass through a food grinder (the preferable method), then place a representative portion in a glass tumbler. If it is necessary to save the cheese and it is not desirable to cut the cheese, in the case of some varieties, take the sample by means of a cheese trier. Remove a plug, as long as possible, from the outer edge, at a point mid-way between the top and bottom surfaces. When possible draw three plugs from different and representative points. For inspection purposes reject one-half inch nearest the rind, but for analyses requiring absolute data reject only such portion at the surface as may be inedible. If a plug sample is used, cut and mix the sample thoroughly in a tumbler by means of a spatula. In the case of some varieties which are very soft, such as cottage and Neufchatel, remove from the package, by means of a spatula, a wedge-shaped portion or a slice as described above, transferring this portion to a tumbler to be mixed thoroughly with a spatula and analyzed at once.

All samples unless analyzed immediately are wrapped in foil or similar

Received for publication June 22, 1939.



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non-absorbent material before being ground and are enclosed in small, air-tight, stoppered containers and analyzed as soon and as rapidly as possible without unnecessary exposure to the air. Samples not analyzed at once are kept in a refrigerator.

2. *Determination of fat:*

(1) Weigh exactly 9 grams of sample as prepared above into a dry, tared, 9-gram, 50 per cent, large-bodied Babcock cream test bottle. This operation should be carried out quickly. The cheese is inserted into the bottle by means of a thin-walled Pyrex glass tube 6 inches long and 11/32 inch outside diameter, and a solid glass rod 6½ inches long and about ¼ inch in diameter, or of the proper size to fit snugly, without binding, into the glass tube.* Cheese is inserted into the glass tube by tapping into the sample with the end of the tube; for very soft cheeses, such as cottage, the tube and the tumbler containing the sample should be held in a horizontal position, while the cheese is being inserted. The outer surface of the tube is wiped several times against the inside of the top of the tumbler and then wiped clean on a paper towel. The tube is inserted into the neck of the bottle and the cheese is pushed into the bottle by means of the glass rod. The rod is wiped clean after it is removed from the tube. By this method cheese is not left sticking to the inner surface of the neck of the test bottle.

(2) Add 12 cc. water at a temperature of 160° to 170° F. Mix well with the cheese.

(3) Add in several installments 17.5 cc. sulphuric acid (sp. gr. 1.82 to 1.83), shaking the bottle after each addition of acid. Let the bottle stand until all particles of cheese have dissolved.

(4) Centrifuge and add water as when testing cream.

(5) Place the bottle in a water bath 130° to 140° F., with the water level above the level of the fat. After 5 minutes add glymol and read the percentage of fat. Duplicate samples should check within 0.5 per cent.

3. *Determination of moisture:*

The sample in a tumbler having been prepared as directed above, the weighing of the sample should be begun immediately and completed as rapidly as possible in order to avoid evaporation due to unnecessary exposure to the air.

(a) For laboratories.

(1) The moisture dishes and covers are dried for 1 hour at 100° C. and allowed to cool for ½ hour in a desiccator containing sulphuric acid or other desiccant.

(2) A cover is placed on the dish and both are weighed on a chemical balance, preferably chainomatic.

* Suggested by Robert E. Hardell, U. S. D. A.

(3) Approximately 2 to 3 grams of the sample are placed in the dish, the cover is immediately replaced, and a second weighing is made. Tests should always be made in duplicate or triplicate for the greatest accuracy.

(4) The samples are dried, with covers, in an oven at 100° C. If a vacuum of 20 inches or more is available, 10 hours' drying should suffice; if no vacuum is used, 24 hours' drying is recommended. Spattering may be minimized by placing the samples in the oven when the oven temperature is below 50° C. so that the samples are heated slowly. Covered dishes must be used to avoid losses of cheese when samples are placed in a hot oven. Vacuum should be applied slowly and released slowly.

(5) After drying, the samples are placed in a desiccator for about one hour, or until they reach room temperature, and each is weighed, without further delay.

(6) Loss in weight divided by weight of sample multiplied by 100 equals percentage of moisture.

Either 30 cc. pyrex beakers or aluminum dishes approximately 50 mm. in diameter and 22 mm. deep may be used. Each dish should be plainly and permanently numbered.

For routine laboratory analysis, if a balance having a tare beam and beams for direct readings and possessing a sensibility reciprocal* of 15 mg. is available, the above procedure is used with the following modifications:

(1) A dish 3 inches in diameter and 1 inch deep is used.

(2) Exactly 10 grams of the freshly-prepared sample are quickly weighed into the dish.

(3) A cover is placed loosely on the dish. After being dried in an oven and cooled in a desiccator, the dish and cover are placed on the balance, and the percentage of moisture is read on the beams to the nearest 0.1 per cent. Duplicate samples when properly dried should check within 0.2 per cent.

(b) For cheese factories.

(1) Tare a dry, aluminum dish with cover on a balance which is equipped with a tare beam and with beams that permit the direct reading of the percentage of moisture. The balance should have a sensibility reciprocal of 15 mg. If the dish is first heated in order to dry it, cooling it afterwards to room temperature is important. A dish 3 inches in diameter and about 1 inch deep is satisfactory.

(2) From the freshly prepared sample weigh 10 grams cheese into the dish. This operation should be done quickly. The lid is placed loosely on the dish so as to permit the escape of moisture. The lid prevents the escape of fat and casein if spattering occurs.

(3) The dish is placed in an oven and heated slowly to a temperature

* The pointer should be deflected a distance equal to one division on the graduated portion when 15 mg. are placed on either scale pan when the scale is loaded to capacity.

of 220° to 230° F. This temperature should be maintained for 24 hours. An electrically heated oven equipped with a heat regulating device is satisfactory for this purpose. If electricity is not available, a pressure steam oven may be used. Since steam is usually not available over a period of 24 hours in the average cheese-factory, with a steam pressure in the jacket of the oven of from 40 to 50 pounds, and a temperature in the oven of 290° F., the drying can be completed in 4 to 6 hours. The temperature should be increased over a period of 1 hour to that desired. This procedure will avoid boiling over of the cheese. Electrically heated ovens can also be used for this short drying treatment.

(4) When the dish is removed from the oven the lid is placed tightly on the dish and the dish with moisture-free material is placed on a cool surface to cool to room temperature. The dish is then placed on the balance and the percentage of moisture read on the beams to the nearest 0.1 per cent after equilibrium has been reached. Duplicate samples should check within 0.2 per cent.

4. *Determination of salt (sodium chloride):*

Weigh accurately approximately 3 grams of the prepared sample of cheese into a 300 cc. Erlenmeyer flask and add 10 cc. of 0.1711 *N* silver nitrate solution, (prepared by using 29.06 grams of C.P. silver nitrate ($AgNO_3$) and making up to one liter in distilled water) or an amount more than sufficient to combine with all of the chlorine. Add 15 cc. of halogen-free, chemically pure nitric acid and 50 cc. of water and boil. As the mixture boils add approximately 15 cc. of saturated potassium permanganate solution in 5 cc. portions. Boil until all cheese particles are digested. Dilute the solution to about 100 cc., decant off the liquid into a beaker, and wash the precipitate by adding 100 cc. of water and decanting again. Add 3 cc. of a saturated solution of ferric ammonium sulfate as an indicator and titrate the excess silver nitrate with 0.1711 *N* potassium or ammonium sulfocyanate (prepared by dissolving 16.63 grams C.P. potassium sulfocyanate and making up to one liter in distilled water). Run a blank on the reagents used, following the same procedure, except to add sugar to destroy the excess of permanganate. The number of cc. of silver nitrate used minus the titration value divided by the weight of sample equals the percentage of sodium chloride in the sample. The reagents should be standardized against a salt solution containing 10 grams of chemically pure, dry sodium chloride per liter.

## REFERENCE

(1) WILSTER, G. H., PRICE, W. V., MORRIS, A. J., GOSS, E. F., AND SANDERS, G. P. Determination of fat, moisture and salt in hard cheese. JR. DAIRY SC. 20: 27. 1937.

# OXIDIZED FLAVOR IN MILK: I. EFFECT OF THE DEVELOPMENT OF OXIDIZED FLAVOR ON THE IODINE NUMBER OF THE PHOSPHOLIPID FRACTION OF MILK

A. M. SWANSON AND H. H. SOMMER
*Department of Dairy Industry, University of Wisconsin*

Oxidized flavor in milk and milk products is generally accepted to be the result of oxidation of one or more of the lipids present in milk. The oxidation of the unsaturated fatty acids present in butterfat was originally thought to be the source of oxidized flavor, but the evidence presented by certain workers shows that the phospholipids present in milk may play an important rôle.

The work reported herein was undertaken to determine which of the lipid constituents in milk had undergone a change on the development of oxidized flavor. The oxidized flavored milk used in these experiments was obtained by the addition of copper in the form of copper sulfate solution. Unfortunately there was no milk available which would develop the off flavor spontaneously.

## REVIEW OF LITERATURE

Thurston (13) states that three classes of milk must be recognized when oxidized flavor is studied. He suggests that the following names and classifications be used: 1, *spontaneous milk*—milk which will develop oxidized flavor without any added metallic catalyst; 2, *susceptible milk*—milk which requires the addition of copper or iron to cause the development of oxidized flavor; and 3, *non-susceptible milk*—milk in which oxidized flavor cannot be produced by the addition of a metallic catalyst. The above classifications will be used in this paper when referring to the different classes of milk.

Evidence was presented by Thurston, Brown and Dustman (14) to indicate that the phospholipid fraction of milk rather than the butterfat was the substance which had undergone oxidation on the development of oxidized flavor. These workers found that the intensity of the oxidized flavor in cream, skimmilk, buttermilk and butter obtained from oxidized milk was in direct relation to their phospholipid content. Remade milk, in which washed cream from oxidized milk was incorporated into normal skimmilk, did not have an oxidized flavor. The addition of copper to remade milk in which butterfat from normal milk was used did not develop an oxidized flavor. Tallowy butterfat incorporated into skimmilk produced a milk having a flavor differing from the typical oxidized flavor. These findings would indicate that the phospholipids which are removed by washing the butterfat play an important rôle in the development of oxidized flavor.

Received for publication August 4, 1939.

Horrall (7) found skimmilk to contain about half the amount of lecithin contained in the original whole milk. Thurston, Brown and Dustman (14) use this fact that skimmilk does contain lecithin to explain the development of oxidized flavor in skimmilk.

Chilson (3), working with spontaneous milk, also found that milk remade from washed cream would not develop an oxidized flavor. The addition of small amounts of copper sulfate to this remade milk would not cause the oxidized flavor to develop, but the addition of 30 p.p.m. of copper produced a tallowy flavor.

Dahle and Palmer (4) substantiate the findings of Thurston, Brown and Dustman (14) in regard to the development of oxidized flavor in susceptible milk, but they found that on the addition of washed cream to skimmilk from spontaneous milk that the remade milk developed an oxidized flavor.

Roland and Trebler (11) standardized milks and after exposing these milks to copper, found that the higher the percentage of fat, the more intense was the oxidized flavor. They also suggest that lecithin may be a factor in the development of oxidized flavor. Horrall (7) found that as the amount of butterfat in milk increased so did the amount of lecithin present increase.

Beck, Whitnah and Martin (1) found no relation between the frequency of occurrence of oxidized flavor and the lecithin content of raw milk.

Thurston, Brown and Dustman (15) observed that homogenization, prolonged agitation at low temperatures, and freezing and thawing of milk caused the milk to become less susceptible to the development of oxidized flavor. The explanation given for the above results is that lecithin is transferred from a film around the fat globules into the serum, and being in a more dilute form and more widely dispersed, it is not so readily oxidized. Tracy, Ramsey and Ruehe (16) and Ross (12) have also found that homogenization makes milk less susceptible to oxidized flavor.

Kende (8) has shown that the iodine number of butterfat decreases with the development of oxidized flavor due to the addition of a metallic catalyst to the milk. He also found that when milks developed the flavor spontaneously there was a decrease in iodine number but not as large as with metal contamination. Dahle and Palmer (4) observed a decrease in the iodine number of butterfat when oxidized flavor developed spontaneously. Brown, Dustman and Thurston (2) could find no appreciable difference due to copper contamination in iodine number of butterfat from normal milk and from oxidized milk.

From the work of Henderson and Roadhouse (5) milk to which copper had been added developed oxidized flavor, but the butterfat had passed only a fraction of its induction period. Holm and Greenbank (6) report that the iodine number of butterfat does not decrease until after the end of the induction period has been reached and the oxygen absorption is on a rapid increase.

## EXPERIMENTAL

The object of these experiments was to compare the iodine numbers of the phospholipid fractions from normal milks with those from oxidized milks and also to compare the iodine numbers of butterfat from the same milks. After some preliminary experiments, the following procedure was adopted for obtaining iodine numbers of the phospholipid fraction.

*Experiment 1*

Ten gallons of milk from the University Creamery intake was pasteurized at 62.2° C. for 30 minutes in two aluminum milk cans. The milk was then immediately cooled to 32° C. and divided into two equal lots. Lot A was separated in a new hand separator and the cream cooled and held at 0° C. To lot B copper sulfate solution was added at a concentration of 3 p.p.m. of copper, and then the milk was cooled to 4° C. and held at that temperature for three days.

The cream from lot A, after being held at 0° C. for 3 hours, was churned in stainless steel laboratory churns. The buttermilk was used as the source of the phospholipid fraction. Four liters of buttermilk were extracted by the Rose-Gottlieb ether extraction method. The ether was then removed from the lipid residue by distillation under vacuum at a temperature of not over 30° C. The distillation was carried on in a complete glass distilling apparatus, and the distillate receiving vessel was packed with dry ice. The vacuum was obtained by means of a mechanical pump.

Immediately upon the removal of the ether from the lipid residue, 500 cc. acetone at 24° C. was added. The phospholipids, lecithin and cephalin, are insoluble in the acetone and form a flocculent white precipitate. The acetone containing the butterfat in solution was removed by decantation. The precipitate was then rewashed with acetone until the acetone remained colorless. The precipitate was dried under vacuum and, when dry, immediately taken up in 60 cc. of chloroform.

Hanus iodine number determinations were made in duplicate on 10 cc. samples of the chloroform solution. The weight of the phospholipids in 10 cc. of the sample was obtained by determination of dry material on evaporation of the chloroform.

The acetone containing the rest of the lipid residue was held at 0° C. for 10 hours. Further precipitation of lipid present in the acetone solution occurred. This precipitate was handled in similar manner to the previous precipitate and duplicate Hanus iodine number determinations were made.

The acetone was also removed from the remaining soluble lipid fraction by means of vacuum distillation and iodine number determinations were made on this residue. A sample of butter oil prepared from the butter obtained in churning was used for Hanus iodine number determinations on butterfat. The peroxide number and the index of refraction were also determined on the last two samples.

The milk in Lot B at the end of the three day storage period had developed a distinct oxidized flavor.

The milk was warmed to 32° C. and separated. The cream was then handled in the identical manner as was the cream in Lot A. Care was taken in the separation and analysis of the lipid fractions so that all conditions were the same.

*Experiment 2*

This experiment was conducted in a manner similar to experiment 1 for the purpose of verifying the previous results. Instead of starting with whole milk, six gallons of raw cream containing 30 per cent of butterfat from the separator in the University Creamery was used. The cream was pasteurized at 62.2° C. for 30 minutes in an aluminum milk can and then cooled to 32° C. The cream was divided into two equal lots. Lot A was immediately cooled to 4° C. and held 10 hours before churning. Three p.p.m. of copper in the form of copper sulfate solution was added to lot B, and it was then cooled to 4° C. and held at this temperature 72 hours before churning.

After churning, the two lots were handled in the identical manner as the two lots in experiment 1, but only the phospholipid fraction was removed from the ether soluble fraction for the iodine number determinations. Hanus iodine number determinations were made on samples of butter oil prepared from the butter obtained on churning of the two lots of cream.

Experiment 1 was conducted during the first week of July 1938 and experiment 2 during the third week of April 1939.

## EXPERIMENTAL DATA

Table 1 summarizes the data obtained in experiment 1. The values for iodine numbers on the different lipid fractions are averages of the duplicate determinations.

TABLE 1

*The effect of the development of the oxidized flavor on the fat constants of the different fat fractions from normal and oxidized milk*

| Lipid fraction | Source of lipid fraction | Fat constants | | |
|---|---|---|---|---|
| | | Iodine No. | Refractive index 40° C. | Peroxide No. |
| Fraction insoluble in acetone at 24° C. (lecithin and cephalin) | Normal milk | 60.34 | | |
| | Oxidized milk | 33.53 | | |
| Fraction soluble in acetone at 24° C. but insoluble at 0° C. | Normal milk | 20.47 | | |
| | Oxidized milk | 32.15 | | |
| Fraction soluble in acetone at 0° C. | Normal milk | 52.58 | 1.450 | 0.0 |
| | Oxidized milk | 52.52 | 1.388 | trace |
| Butter-oil | Normal milk | 47.96 | 1.419 | 0.0 |
| | Oxidized milk | 48.50 | 1.418 | trace |

Table 2 gives a summary of the iodine number determinations made on the phospholipid fraction (lecithin and cephalin) of normal and oxidized milks and on the butter-oil samples obtained from the same milks in experiment 2.

TABLE 2

*The effect of the development of oxidized flavor on the iodine numbers of the phospholipid fractions and of samples of butter-oil from normal and oxidized milk*

| Lipid fraction | Source of lipid fraction | Iodine number |
|---|---|---|
| Fraction insoluble in acetone at 24° C. | Normal cream | 48.72 |
| | Oxidized cream | 33.67 |
| Butter-oil | Normal cream | 33.12 |
| | Oxidized cream | 32.82 |

## DISCUSSION

Evidence in the literature suggests that the lecithin in milk rather than the butterfat becomes oxidized during the development of oxidized flavor. No chemical evidence has been given to substantiate the above assumption outside of the work of Brown, Dustman and Thurston (2). These workers could find no appreciable difference in the iodine number of butterfat from normal milk as compared to butterfat from oxidized milk. They also suggest that if oxidation of the lecithin had occurred there would not have been a marked change in the iodine number of the butterfat due to the small quantity of lecithin present. From the work of Kende (8) and Dahle and Palmer (4) the butterfat must also undergo oxidation when the oxidized flavor develops spontaneously in milk to which no metallic catalyst has been added.

The phospholipids, lecithin and cephalin, are insoluble in acetone at room temperature and will form a white flocculent precipitate, while the rest of the lipid fraction is quite soluble. This property of the phospholipids, lecithin and cephalin, was used in removing them from ether soluble residues obtained from normal and oxidized buttermilk. Lecithin and cephalin are soluble in chloroform, and chloroform solutions of the phospholipids were used for the Hanus iodine number determinations.

In this work no attempt was made to separate the cephalin from the lecithin, so the reported results indicate changes in the phospholipid fraction containing lecithin and cephalin. The results may then be due to oxidation occurring in the lecithin or in the cephalin or in both substances. Kurtz, Jamieson and Holm (9) by means of titration with sodium hydroxide found that purified glycero-phosphatides of milk consist of 44 per cent cephalin and 56 per cent lecithin. Rewald (10) in a recent paper found butter phosphatides have approximately the following composition: 36 per cent cephalin (alcohol insoluble), 50 per cent lecithin (alcohol soluble) and a

14 per cent fraction which is soluble in hot alcohol and insoluble in cold alcohol.

Brown, Dustman and Thurston (2) calculated the theoretical iodine number of a lecithin which contains one saturated fatty acid, stearic, and one unsaturated fatty acid, oleic, to be 31.54. At one time it was generally assumed that milk lecithin contained one saturated and one unsaturated fatty acid; this assumption has since been found to be untrue. Kurtz, Jamieson and Holm (9) assumed that there is a mixture of fatty acids in milk phospholipids and that oleic acid represents 70.0 per cent of the fatty acids present. They also state that there are none of the lower fatty acids in the glycero-phosphatides of milk.

From experiments 1 and 2 the iodine numbers of the phospholipid fraction from oxidized milk were 33.53 and 33.67 respectively. The oxidation of the phospholipid fraction is not complete and the final iodine numbers are quite close to the theoretical iodine number of 31.54 for lecithin assuming stearic acid and oleic acid, so it would seem that one oleic acid molecule in each molecule of lecithin remained unoxidized. The iodine number of the phospholipid fraction from the normal summer milk in experiment 1 was 60.34 and for the normal early spring milk in experiment 2 was 48.72. Both of these iodine numbers were considerably higher than those for phospholipid fractions of oxidized milk, which indicates the presence of more than one unsaturated fatty acid in each molecule of lecithin. The phospholipid fraction from summer milk contains more unsaturated fatty acids than the phospholipid fraction from early spring milk. Apparently the presence of natural reducing substances in summer milk tends to inhibit the development of oxidized flavor even though the iodine number is higher.

In both experiment 1 and experiment 2, the milk to which copper had been added developed typical oxidized flavor. In experiment 1 there was a 44.41 per cent reduction in iodine number of the phospholipid fraction, and in experiment 2, a 30.89 per cent reduction in iodine number of the phospholipid fraction on the development of oxidized flavor.

There was no significant difference between the iodine numbers obtained on samples of butterfat from normal and oxidized milk. In experiment 1 there also appeared little difference in the refractive indices of butterfat from normal and oxidized milk. The sample of butterfat from oxidized milk showed a trace of peroxide formation but none was observed in the sample of butterfat from normal milk.

In experiment 1 the lipid fraction soluble in acetone at 24° C. and insoluble at 0° C. evidently contains some of the fats, particularly the ones containing saturated fatty acids because the iodine number is lower than for the regular butterfat. Fats are known to be insoluble in acetone at low temperature. The fraction from normal milk is lower in iodine number

than the fraction from oxidized milk. This difference may have a significant relation to oxidized flavor development but no explanation is apparent.

The lipid fraction soluble in acetone at 0° C. showed little difference in iodine number between that obtained from normal milk and oxidized milk. The sample from oxidized milk showed a slight decrease in refractive index and a trace of peroxide formation. The decrease in refractive index of the fraction from oxidized milk may also have significant relation to the development of the oxidized flavor.

These experiments were conducted in a dark room to minimize the effect of light on the phospholipid fractions, which are known to be readily oxidized when exposed to light and air. Small samples of the phospholipid fraction from normal and oxidized milk were dried and then observed for appearance. The sample from normal milk was white and flaky on drying, but the sample from oxidized milk had a greenish tint and was greasy in appearance. Neither sample had an odor or taste which resembled the oxidized flavor of milk.

## SUMMARY AND CONCLUSIONS

The Hanus iodine numbers of the phospholipid fractions from normal and oxidized milk were determined. The development of oxidized flavor is accompanied by a marked decrease in the iodine number of the phospholipid fraction. Iodine number determinations on samples of butterfat from normal and oxidized milk showed no significant difference. The conclusion can be drawn that the development of oxidized flavor in milk, catalyzed by copper, is primarily due to the oxidation of the phospholipid fraction.

The oxidation of the unsaturated fatty acid in the phospholipid fraction is not complete, but the indications are that one molecule of an unsaturated fatty acid, undoubtedly oleic acid, remains unoxidized.

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# EFFECT OF CONDENSING ON THE DEVELOPMENT OF OXIDIZED FLAVOR

W. J. CORBETT AND P. H. TRACY
*Department of Dairy Husbandry, University of Illinois, Urbana, Illinois*

In previous work done at this station by Tracy (1) on the manufacture of bottled concentrated whole milk, it was observed that the oxidized flavor which frequently occurred in regular milk did not develop in the concentrated milk, even though no effort was made to exclude air from the samples after condensing. In 1938, Guthrie, Hand and Sharp (2) reported that the destruction of ascorbic acid and the development of oxidized flavor could be largely or completely prevented by the removal of oxygen from the milk by treating under vacuum. The following study was made to determine more exactly the role of the condensing process in the prevention of the oxidized flavor.

## METHODS

All samples of whole milk were standardized to four per cent fat and then pasteurized by heating to 143° F. for 30 minutes in a stainless steel vat. The milk was condensed in either a small laboratory glass condensing unit or in a three-foot stainless steel condensing pan.[1] The condensing was done at a vacuum of 24–25 inches at 135–140° F. By means of a double valve arrangement in the pan outlet it was possible to take samples during the condensing process without shutting down. Samples were cooled immediately to approximately 40° F. and later reconstituted to four per cent fat with tap water. To a part of the samples 3 p.p.m. of copper were added in a 1 per cent solution of copper sulfate. The milk samples were held at 40° F. and judged for degree of oxidized flavor at various time intervals.

## CONDENSING AS A RETARDER OF OXIDIZED FLAVOR

Several lots of four per cent fat milk were pasteurized and condensed to a 2–1 concentration. The effectiveness of condensing as a retarder of oxidized flavor is shown by the data in table 1, which is typical of many trials.

The data show conclusively that the reconstituted condensed milk did not develop the oxidized flavor, even when 3 p.p.m. of copper was added. In these trials no precautions were taken to exclude air from the condensed milk after it was taken from the vacuum pan. In several experiments compressed air was bubbled through the reconstituted condensed milk to which copper had been added for several minutes, and yet the oxidized flavor did not develop during holding periods of several days.

Received for publication September 5, 1939.

[1] Although all the data given in this paper were taken from experiments in which the large vacuum pan was used, substantially the same results were obtained with the small laboratory condensing unit.

TABLE 1

*Effectiveness of condensing in retarding the development of oxidized flavor*

| Sample | Degree of oxidized flavor after: | | |
|---|---|---|---|
| | 1 day | 2 days | 3 days |
| 1. Pasteurized—unconcentrated (4% fat) | – | – | – |
| 2. Pasteurized—unconcentrated (4% fat) plus 3 p.p.m. copper | +++½ | +++++ | ++++++ |
| 3. Reconstituted condensed milk (4% fat) | – | – | – |
| 4. Reconstituted condensed milk (4% fat) plus 3 p.p.m. copper | – | – | – |

AMOUNT OF CONDENSING NECESSARY TO RETARD OXIDIZED FLAVOR

Twelve hundred pounds of 4 per cent fat milk were pasteurized and drawn into the vacuum pan to determine how much condensing was necessary to protect the samples against the development of oxidized flavor. Samples of the partially condensed milk were taken at 15-minute intervals during the condensing process. The results are given in table 2.

TABLE 2

*Relation of extent of concentration to development of oxidized flavor in reconstituted, condensed milk*

| Sample | Fat test before reconst. | Degree of oxidized flavor after: | | | | | |
|---|---|---|---|---|---|---|---|
| | | 1 day | | 2 days | | 3 days | |
| | | No cu | 3 p.p.m. cu | No cu | 3 p.p.m. cu | No cu | 3 p.p.m. cu |
| 1. Past. | 4.0 | – | +++ | – | +++++ | – | ++++++ |
| 2. Cond. 15 min. reconstituted to 4% fat | 5.0 | – | – | – | – | – | ++½ |
| 3. Cond. 30 min. reconstituted to 4% fat | 6.05 | – | – | – | – | – | ± |
| 4. Cond. 50 min. reconstituted to 4% fat | 8.50 | – | – | – | – | – | – |
| 5. Cond. 50 min. Homogenized at 2500 pounds pressure, reconstituted to 4% fat | 8.50 | – | – | – | – | – | – |

It is apparent that rendering the condensed milk immune from developing the oxidized flavor involves more than simply a removal of oxygen as one would expect the dissolved air to be removed within a few minutes after the condensing process was started. As shown in these trials it is necessary to condense to practically a 2–1 concentration to entirely protect the milk from developing an oxidized flavor, suggesting that a physical change in the

serum constituents may be responsible for the protection of the fat against oxidation.

### MILK MADE FROM CREAM AND CONDENSED SKIMMILK IMMUNE TO OXIDIZED FLAVOR

The following experiment was performed to determine if condensing skimmilk would be effective in preventing an oxidized flavor in milk made from cream and condensed skimmilk. Skimmilk was pasteurized by heating to 143° F. for 30 minutes and then concentrated to 30 per cent solids. Four per cent fat milk was made from 32 per cent cream, and condensed skimmilk and water. A control sample containing 4 per cent fat was made from the same lots of 32 per cent cream and the skimmilk. The results are given in table 3.

TABLE 3

*Effect of concentrating the plasma portion of milk upon oxidized flavor development*

| Sample | Degree of oxidized flavor after: | | |
|---|---|---|---|
| | 1 day | 2 days | 3 days |
| 1. 4% milk made from cream and skimmilk | – | – | ++ |
| 4% milk made from cream and skimmilk +3 p.p.m. Cu | +½ | ++++ | ++++ |
| 2. 4% milk made from cream, condensed skimmilk and water | – | – | – |
| 4% milk made from cream, condensed skimmilk and water +3 p.p.m. Cu | – | – | ± |

These data show that the effect of the condensing process in retarding oxidized flavor development is upon the plasma portion of the milk. It is possible that the condensing process causes a shift in the oxidation-reduction potential to the reduced side by the liberation of reducing substances or by the liberation of substances which act as antioxidants.

### EFFECT OF CONDENSING ON THE CURD TENSION OF MILK

There are several instances in which factors which retard or prevent tallowy flavors in milk also reduce the curd tension of the milk. Examples of this are homogenization, addition of certain enzymes, addition of sodium salts, and high heat treatment.

Curd tension measurements were made on the samples of reconstituted condensed milk to determine the effect of condensing on the hardness of the curd. The tentative procedure adopted by the American Dairy Science Association Subcommittee on Curd Tension Measurements in 1938 was followed, and a Submarine Signal curd tension machine was used to make the measurements. The results are given in table 4. Condensing either whole or skimmilk and then reconstituting to the original solids content lowered the curd tension. The more condensing the milk was subjected to, the greater was the influence on the curd tension.

TABLE 4

*Effect of condensing on the curd tension of milk (condensed milk reconstituted with water to 4 per cent fat)*

| Sample | Fat test before reconstituting | Curd tension on reconstituted milk (grams) |
|---|---|---|
| **Part A. Using condensed whole milk** | | |
| 1. Pasteurized | 4.0 | 43.5 |
| 2. Cond. 15 min. | 5.0 | 45.0 |
| 3. Cond. 30 min. | 6.05 | 40.5 |
| 4. Cond. 50 min. | 8.5 | 35.0 |
| 5. Same as 4.—Homogenized at 2500# pressure | 8.5 | 22.0 |
| **Part B. Using condensed skimmilk plus cream** | | |
| 1. Past. skimmilk (9.2% T.S.) | | 55 |
| 2. Cond. skim 30% T.S. reconstituted to 9.2% T.S. with water | | 33 |
| 3. 32% cream and skimmilk to make 4% fat milk | | 36 |
| 4. 32% cream and cond. skimmilk and water to make 4% fat milk | | 22 |

EFFECT OF CONDENSING ON THE RATE OF DESTRUCTION OF ASCORBIC ACID

Several investigators have shown that the oxidation of ascorbic acid (Vitamin C) in milk precedes that of fat oxidation. Guthrie, Hand and Sharp (2) have shown that evacuating hot milk and the subsequent storing in bottles under a vacuum prevents the destruction of ascorbic acid, as well as the development of oxidized flavor.

The following experiment was performed to determine the effect of condensing on the destruction of ascorbic acid. Twelve hundred pounds of 4 per cent fat milk were pasteurized at 143° F. for 30 minutes and condensed under a vacuum of 24–25 inches to a concentration of 8.2 per cent fat. Samples were obtained 20 minutes after the condensing process had started and again 25 minutes later when the milk had been concentrated 2 to 1. The relation of condensing to the oxidation of ascorbic acid and the development of the oxidized flavor are shown by the data in table 5. Sharp's (3) procedure was followed for determining the ascorbic acid content.

Apparently there is no relation between the oxidation of ascorbic acid and the development of oxidized flavor in reconstituted condensed whole milk. In the case of the control milk containing copper and the 2–1 condensed milk reconstituted to 4 per cent fat to which copper was added, the ascorbic acid was oxidized at approximately the same rate; however, the control milk developed a strong oxidized flavor, and the condensed reconstituted milk was entirely free from oxidized flavor during the entire holding period. If, as Sharp contends, there is a relationship existing between the oxidation of ascorbic acid and the development of the oxidized flavor, the mere absence of the ascorbic acid does not mean that the oxidized flavor will necessarily develop.

TABLE 5

*Effect of condensing on rate of destruction of ascorbic acid and development of oxidized flavor*

| Sample | Per cent fat | Ascorbic acid content (mgs. per liter) | | | | Oxidized flavor after | | |
|---|---|---|---|---|---|---|---|---|
| | | Fresh | 1 day | 2 days | 3 days | 1 day | 2 days | 3 days |
| Regular pasteurized milk | 4.0 | 18.2 | 14.7 | 12.8 | 10.3 | – | – | – |
| Past. + 3 p.p.m. Cu | 4.0 | 18.2 | < 1.0 | < 1.0 | < 1.0 | +++ | +++++ | +++++ |
| Cond. 20 min. | 5.5 | 18.9 (13.7)* | 6.60 (4.80) | 1.1 | < 1.0 (< 1.0) | – | – | – |
| Cond. 20 min. + 3 p.p.m. Cu | 5.5 | 18.9 (13.7) | 1.38 (1.00) | 1.66 (1.22) | 1.25 (< 1.0) | – | +++ | +++ |
| Cond. 20 min. and reconst. to 4% fat | 4.0 | 13.8 | 7.48 | 1.43 | < 1.0 | – | – | – |
| Cond. 20 min. and reconst. to 4% fat + 3 p.p.m. Cu | 4.0 | 13.8 | < 1.0 | < 1.0 | < 1.0 | ± | ± | + |
| Cond. approx. 2–1 | 8.2 | 36.4 (16.8)* | 34.3 (15.9) | 28.1 (13.0) | 20.8 (9.65) | – | – | – |
| Cond. 2–1 + 3 p.p.m. Cu | 8.2 | 36.4 (16.8) | 12.0 (5.5) | 10.4 (4.82) | 6.97 (3.23) | – | – | ± |
| Cond. 2–1 and reconst. to 4.0% fat | 4.0 | 16.6 | 13.20 | 9.03 | 6.45 | – | – | – |
| Cond. 2–1 and reconst. to 4.0% fat + 3 p.p.m. Cu | 4.0 | 16.6 | < 1.0 | < 1.0 | < 1.0 | – | – | – |

* Numbers in parentheses refer to values calculated on a 4% fat basis.

## POSSIBLE EXPLANATION FOR EFFECT OF CONDENSING PROCESS ON THE RETARDATION OF AN OXIDIZED FLAVOR

As has been mentioned previously, there are several instances in which factors which affect the milk plasma, as shown by reductions in curd tension, also retard the development of oxidized flavor. It is entirely possible that these factors which lower the curd tension also tend to partially break down the milk protein. Possibly some of the amino acids which act as antioxidants and prevent the development of oxidized flavor are liberated during these processes. Corbett and Tracy (4) have studied the antioxidative effect of several amino acids or their esters, and found tyrosine, tyrosine ethyl ester, leucine N-amyl ester and glutamic diethyl ester to be effective antioxidants in milk. Skimmilk powder has also been found to have antioxidative properties. The results of this study are not in agreement with those of Guthrie, Hand and Sharp (2), who explain the effect of vacuumizing milk in retarding oxidized flavor development as one of oxygen removal. It seems that the explanation needs to take into consideration possible changes in the protein complexes of the plasma portion rather than the physical removal of air or oxygen, since the introduction of air after condensing does not cause the development of the oxidized flavor. Additional studies are being made to obtain further proof for this hypothesis.

## CONCLUSIONS

1. Condensing milk under vacuum to a concentration of approximately 2–1 was found to prevent the development of oxidized flavor in both the condensed milk and condensed milk reconstituted to the original solids concentration.

2. Four per cent fat milk made from condensed skimmilk and 32 per cent cream did not develop an oxidized flavor, even when as much as 3 p.p.m. of copper were added.

3. The effect of condensing in retarding oxidized flavor development is thought to be due to the liberation in the serum portion of the milk certain antioxidative constituents that are probably derivatives of the milk protein.

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# THE INFLUENCE OF "WHITE-METAL" COPPER-NICKEL ALLOYS ON THE FLAVOR OF MILK

J. L. HENDERSON AND C. L. ROADHOUSE
*Division of Dairy Industry, University of California, Davis*

As shown by investigations at many experiment stations, copper contamination from dairy equipment is the principal cause of oxidized flavor in milk; furthermore, chrome-nickel iron alloy or stainless steel of the 18–8 series is not corroded by milk, and its use is desirable if oxidized flavor from metal sources is to be avoided.

Since technical difficulties have been encountered in fabricating certain parts of dairy equipment from stainless steel, manufacturers have sought alloys more easily cast and machined. This has been particularly true in the making of certain fittings, valves, and bearings. Most of the alloys developed have copper and nickel as their base, and several other elements have been added to give the alloy the desired properties—namely, the ability to make a sound casting, to be machined easily, to remain bright after use, and to leave unimpaired the flavor of milk. Copper-nickel alloys have been used in the dairy industry for many years, and those previously studied have been found to cause oxidized flavor (1, 2). Some of the "white-metal" copper-nickel alloys recently developed include the addition of other elements that apparently modify their corrosion rate in milk. The present study reports tests conducted with certain of these alloys.

TABLE 1

*Composition of the alloys tested*

| Alloy No. | Percentage composition | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | Copper | Nickel | Tin | Lead | Zinc | Iron | Manganese | Chromium |
| A | 65.6 | 31.0 | .... | 2.0 | .... | Trace | 1.0 | 0.5 |
| B | 65.0 | 20.0 | 8.0 | 2.0 | 3.0 | 2.0 | .... | .... |
| C | 62.0 | 20.0 | 8.0 | 2.0 | 3.0 | 2.0 | .... | 3.0 |
| D | 66.0 | 20.0 | 3.0 | 4.0 | 4.0 | 2.5 | 0.5 | .... |
| E | 63.0 | 20.0 | 3.0 | 4.0 | 4.0 | 2.5 | 0.5 | 3.0 |
| F | 66.5 | 31.0 | .... | 2.0 | .... | Trace | Trace | 0.5 |
| G | 65.5 | 31.0 | .... | 2.0 | .... | Trace | 1.0 | 5.0 |

## EXPERIMENTAL PROCEDURE

*Source of milk.* The milk used in these studies was obtained from normal cows of the Station herd producing milk of good flavor. All the cows received identical rations. The milk was drawn into new, well-tinned pails

Received for publication August 12, 1939.

and was poured immediately into amber bottles in ice water. All surfaces of the pails and glass utensils coming in contact with the milk were rinsed with distilled water to prevent the addition of copper from tap or boiler water.

*Determination of corrosion rate.* The milk was pasteurized in contact with thin strips of the cast alloys 1 inch by 2 inches in size. These were cleaned, dried, and weighed before and after pasteurization according to the procedure previously described by Guthrie, Roadhouse, and Richardson (2). The alloys were agitated in the milk during the heating, holding, and cooling procedures, which required approximately 1½ hours. The pasteurization temperature was 142° F.

The averages of the corrosion rates reported in table 2 are for eight determinations.

*Scoring the milk.* The processed samples, together with control samples of the same milk, were held at 40° F and scored for flavor by two judges after storage periods of two to four days. The controls and test samples were arranged in random order in duplicates and scored as unknowns. Milk samples reported as having definitely oxidized flavor were detected by both judges in the duplicate samples.

*Determination of ascorbic acid.* Ascorbic acid was determined by a modified Bessey and King titration. The acid solution used for precipitating the protein and for controlling the pH of the serum to be titrated consisted of 15 per cent trichloracetic acid plus 2 per cent meta-phosphoric acid. First 15 ml. of the acid solution was added to 25 ml. of milk. After standing for a few minutes with frequent shaking, the mixture was filtered. Then 10 ml. of the clear serum was titrated with approximately 0.2 per cent sodium 2.6 dichlorobenzenone indophenol solution held in a micro burette. The end point was the first permanent light-pink color that persisted for 30 seconds as determined by a stop watch. The dye was standardized by the thiosulfate method of Menaker and Guerrant (3). The first ascorbic-acid determinations were made immediately after the completion of the experimental treatment of the milk, and subsequent determinations followed various storage periods at 40° F.

## EXPERIMENTAL DATA

*Rates of corrosion.* Typical data secured on the rates of corrosion of the alloys by milk during the pasteurization process appear in table 2. The weight losses of the alloys obtained from eight tests are presented in table 3. The losses are reported in milligrams per square decimeter per day in order that comparisons may be made with values reported, in the literature, for other metals. Alloys A, F, and G were noticeably tarnished after exposure to milk, whereas the others remained bright.

*Influence on milk flavor.* In studying the development of copper-induced

oxidized flavor, one must consider the differences in susceptibility of the milk produced by the individual cows to develop this flavor defect. Eight cows were used in this experiment; and from other experiments not reported in this paper, five were classified as "susceptible" and three as less "susceptible" to the development of copper-induced oxidized flavor. The "susceptible" cows produced milk that developed the flavor when minute quantitites of cupric ion were present, whereas the less susceptible ones required appreciable amounts of cupric ion (approximately 0.5 p.p.m.). The variation in the susceptibility of milk of individual cows to develop oxidized flavor

TABLE 2

*Variation in the susceptibility of milk of different cows to develop oxidized flavor*

| Cow No. | Alloy sample No. | Days stored at 40° F. | | | Mlg. weight loss during pasteurization |
|---|---|---|---|---|---|
| | | 2 | 3 | 4 | |
| 181 | Control | − | − | − | ..... |
| | A | + | + | + | 2.18 |
| | B | − | + | + | 0.60 |
| | C | − | + | + | 0.74 |
| | D | − | + | + | ..... |
| | E | − | + | + | 2.43 |
| | F | + | + | + | 2.40 |
| | G | + | + | + | 2.83 |
| 498 | Control | − | − | − | ..... |
| | A | − | ± | + | 2.79 |
| | B | − | − | − | 0.39 |
| | C | − | − | − | + 0.17 |
| | D | − | − | − | 0.86 |
| | E | − | − | − | 2.20 |
| | F | − | ± | + | 1.40 |
| | G | − | ± | + | 3.24 |

− No oxidized flavor or odor.
+ Oxidized flavor or odor.
± Judges not in agreement as to flavor.

TABLE 3

*Corrosion rates and influence of alloys on flavor of milk*

| Alloy No. | Average of eight trials | | |
|---|---|---|---|
| | Weight loss calculated as mlg/dm²/day† | Percentage* of samples having oxidized flavor after storage at 40° F. | |
| | | 2 days | 3 days |
| A | 117.0 | 87.7 | 100.0 |
| B | 26.6 | 37.5 | 62.6 |
| C | 14.2 | 37.5 | 62.6 |
| D | 29.4 | 37.5 | 50.0 |
| E | 143.0 | 37.5 | 62.6 |
| F | 114.0 | 75.0 | 100.0 |
| G | 136.0 | 87.7 | 100.0 |

* 5 susceptible cows, 3 less susceptible cows.
† milligram per square decimeter per day.

when exposed to copper-nickel alloys of different compositions is illustrated in table 2. Cow 181 is classified as susceptible, whereas cow 498 is less susceptible. The percentages of samples having oxidized flavors after 2 and 3 days' storage at 40° F are shown in table 3. Here again are illustrated the differences in susceptibility of the milks. Sufficient copper went into solution from alloys A, E, and F to exceed the copper-threshold value of even the less susceptible milks.

*Ascorbic acid oxidation.* How temperature and the length of time of exposure of the copper-nickel alloys to milk affect the rate of ascorbic acid oxidation is shown in figure 1. The milk exposed to alloy D for 1½ hours showed a very slight oxidized flavor after 3 days, whereas that exposed to G had a very strong oxidized flavor. The other milks did not show this defect.

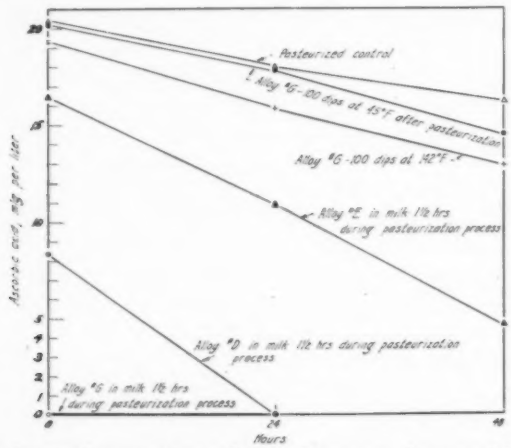


FIG. 1. Influence of temperature and time of exposure of copper-nickel alloys on the rate of oxidation of ascorbic acid in milk.

To arrive at an approximation of the amount of copper going into solution when the alloys were exposed to milk for brief periods of time, 0.1, 0.01, and 0.001 p.p.m. of cupric ion were added to other portions of the same milk into which alloys D and G were dipped 100 times at 142° F. The ascorbic-acid contents of the milk were determined immediately after treatment and after 2 and 4 days' storage at 40° F (figure 2). The milks were also scored for flavor. The raw and pasteurized samples to which 0.1 p.p.m. cupric ion were added had a very slight oxidized flavor on the fourth day, whereas the others were not influenced.

Strips of pure nickel, zinc, and lead measuring 1 inch by 2 inches were also agitated in milk during the pasteurization process. The rate of oxidation of ascorbic acid was not influenced by this amount of exposure to these metals, nor was oxidized flavor detected in any of the milks so treated.

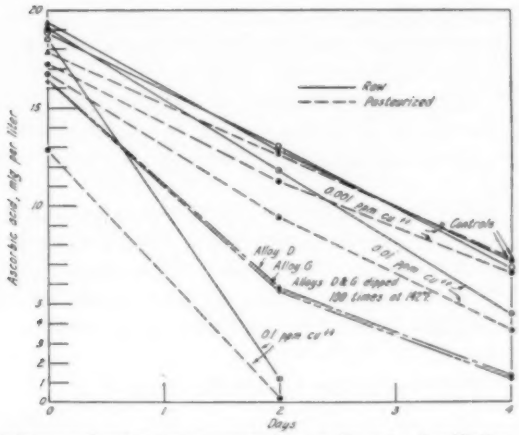


FIG. 2. Influence of cupric ion concentration on the rate of oxidation of ascorbic acid in milk.

## DISCUSSION OF RESULTS

As is indicated by the variations in the corrosion rates of cast copper-nickel alloys and their influence on the flavor of milk, one must test each alloy experimentally in order to evaluate the desirability of its use in fabricating milk-plant equipment. A study of table 3 reveals that alloys A, E, F, and G have high corrosion rates in milk as compared with alloys B, C, and D. According to table 1, alloys A, F, and G are similar in composition in that all contain 31 per cent nickel and no tin or zinc. Alloy E, however, has a composition more nearly like that of B, C, and D, since it contains 20 per cent nickel and also tin and zinc. By analogy one would expect alloy E to have a corrosion rate more nearly like C than G. The effect on milk flavor (table 3), however, shows that E caused oxidized flavor to develop only in the more "susceptible" milks, as did B, C, and D. Figure 1 shows also that alloy E had even less effect on ascorbic acid oxidation than did D. According to these results, copper was less soluble in alloy E than in G despite their similar corrosion rates. The conclusion is that the solubility of the elements in these alloys is a complex function of their compositions, their alloy structures, or a combination of these.

When the alloys were in contact with the milks during the entire pasteurization process, alloys B, C, and D were most satisfactory as judged from the rate of corrosion and the influence on milk flavor. Alloys B and C are reported to be very difficult to machine, and the cost of the added tin over that contained in D would not be justified. Alloy D is considered the most satisfactory for commercial use.

A brief exposure of any of the alloys to milk such as would result from passing milk through fittings in a stainless-steel pipeline would not be ex-

pected to permit sufficient copper to go into solution to cause oxidized flavor to develop in mixed milks. The evidence for this is the effect on ascorbic acid destruction after the alloys were dipped 100 times in the milks at 45° and at 142° F. Judging, however, from the rates of corrosion for a longer exposure period, from the appearance of the metals after exposure to milk, and from their influence on the flavor, alloy D is the most satisfactory of the alloys studied.

The flux used in preparing the alloy at the foundry is said to affect the corrosion resistance. It is used in the molten metal to serve as a deoxidizer to remove sulfur and oxygen. The flux is finally removed from the molten alloy as slag. For the copper-nickel alloys, pure magnesium in stick form has been recommended at the foundry in which the samples studied were prepared.

## SUMMARY AND CONCLUSIONS

Several copper-nickel alloys have been studied to determine the rates of corrosion in milk, together with the effect upon milk flavor and upon ascorbic acid destruction. The following conclusions have been drawn:

1. Less copper went into solution from the alloys containing tin and zinc, so that the flavor of the milk was less influenced than with alloys in which these elements were absent.

2. In general the rates of corrosion in milk were lower with alloys containing tin and zinc. The influence on ascorbic acid destruction, however, was found to be a more reliable index of the probable effect of the alloys on milk flavor. The effect on ascorbic acid destruction is a specific test for copper in solution: whereas the loss in weight does not necessarily indicate the relative amount of copper going into solution.

3. Nickel, lead, and zinc did not influence the oxidation of ascorbic acid and did not cause oxidized flavor in the milks pasteurized in contact with pure strips of these elements.

## ACKNOWLEDGMENTS

The authors have appreciated the cooperation of Mr. Loomis Burrell of the Cherry-Burrell Corporation in arranging for certain of the alloys used in the study and for the composition of the alloys. Acknowledgment is also made of Mr. George Young's assistance with some of the experiments.

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# WATER SORPTION BY DRY MILK SOLIDS. III. A COMPARISON OF RESULTS OBTAINED BY THE CRYOSCOPIC, VAPOR PRESSURE, AND VOLUME CONTRACTION METHODS

E. L. JACK
*University of California*

The papers of the present series (1, 2, 3) have reported investigations on the effect of various treatments on the water-solids relationships in dry milk. These studies have been made using the volume contraction method as previously described. Other investigators have reported data on particular phases of the subject using the cryoscopic method and the vapor pressure method. It is of interest to compare results obtained by each of these methods on the same samples and to consider possible reasons for any disagreement among them.

## REVIEW OF LITERATURE

The cryoscopic method for the determination of "bound" water was devised by Newton and Gortner (4). The principle of the method depends upon the use of a dehydrating agent to take up the "free" water and then determining the freezing point of this solution of dehydrating agent and free water. When part of the total water present is "bound," a subnormal lowering of the freezing point results and the percentage of water not removed by the dehydrating agent can be readily calculated. In actual practice one mole of sucrose per liter of total water in the system is commonly used, although Briggs (5) has found that ethyl alcohol is more satisfactory in certain systems.

This method has had extensive application to biological materials and has been used to study the "bound" water content of dairy products. Pyenson and Dahle (6) have investigated the ability of dried skimmilk, prepared by the vacuum drum process to "bind" water. Their results showed that the dry material "binds" water to the extent of 60.1 per cent of its dry weight, when freshly prepared and approximately 40 per cent when four to eight weeks old.

The vapor pressure method depends upon the lesser fugacity of water in the sorbed state than in the free state. Sorbed water is not free to vaporize to the same extent as free water, and consequently does not exert its normal pressure. The relative vapor pressure is related to the degree of sorption as expressed by the water content of the system.

The vapor pressure method gained attention through the classic work of van Bemmelen (7) on silica gels. It has since been used in many fields of investigation. Supplee (8) was the first to show the equilibrium relationship between water vapor and water sorbed by milk powder. His results

Received for publication August 17, 1939.

show that milk powder will take up about 14 per cent of its weight of water at 80 per cent relative humidity. Recently, Davis (9), using the same technique found that the moisture content of dried skim milk was about 17–18 per cent when in equilibrium with an atmosphere of 80 per cent relative humidity.

The volume contraction method employs the increase in density that accompanies sorption to measure the degree of sorption. Because the density of the system increases when sorption occurs there is a corresponding decrease in volume which can readily be determined in a dilatometer at constant temperature. The method has been used by various investigators (10, 11, 12, 13, 14) to study different materials but not previously for dry milk solids.

Several comparisons have been made of the different methods for determining the sorbed water—free water equilibrium. Sayre (15) compared the cryoscopic, calorimetric, and dilatometric (based on the freezing expansion coefficient of water) methods for gum arabic solutions and obtained reasonably good agreement for the averages, but he concluded that the cryoscopic method was the least reliable of the three. He pointed out that these methods yield results only at the freezing point. It has been generally recognized that the vapor pressure method does not yield values as great as the cryoscopic technique. Briggs (5, 16) has compared the two methods as has Grollman (17). Grollman, particularly, has criticised the usual equation used for calculations with the cryoscopic method as not being applicable when other solutes are present.

## EXPERIMENTAL PROCEDURE

The cryoscopic technique was carried out as recommended by Newton and Gortner (4), measuring the freezing point depression caused by dissolving a mole of sucrose in a liter of water containing 10 per cent of dry milk solids which had been in solution 24 hours. The apparatus used was a Hortvet cryoscope and a previously standardized Beckman thermometer. The usual precautions with respect to super cooling were observed.

Aqueous vapor pressures of the samples at definite water contents were measured with a modified Regnault dew-point hygrometer. The apparatus consists of a 250 ml. Ehrlenmyer flask containing the sample having a known water content. Suspended within the flask through a tightly fitting stopper is a metal tube with a highly polished metal mirror soldered to its side. This apparatus is maintained at constant temperature, in this case at 20° C., until equilibrium is established between the sample and the atmosphere within the flask. Then ethyl ether or other suitable refrigerant is poured into the metal tube, a thermometer and an aspirator tube inserted and air bubbled through the ether with an atomizer bulb until fog appears on the mirror; the temperature is noted at this time and again when the mirror clears. The average of the two is considered the dew point of the atmos-

phere in equilibrium with the sample. Vapor-pressure-temperature tables give the vapor pressure in millimeters of mercury at saturation at various temperatures. With experience and care it is possible to observe fogging and clearing of the mirror within about a 0.2° C. interval. Comparison of this procedure with that outlined by Wilson (18) using sulphuric acid solutions to control humidity showed them to give similar results up to about 80 per cent relative humidity; above this humidity neither is satisfactory because of the necessity for extremely sensitive temperature control and the length of time necessary for equilibrium to be established. Supplee (8) and Davis (9) have used sulphuric acid solutions to control humidity.

Volume contraction was measured as previously described (1, 2, 3) at 20° C.

The samples studied consisted of eight different lots of dry milk solids, four prepared by the atmospheric roll process and four by the spray process from skimmilk which had been preheated at 71.1° C. (160° F.), 76.6° C. (170° F.), 82.2° C. (180° F.), and 93.3° C. (200° F.) for 30 minutes for each process.

## RESULTS

The results of these studies have been calculated and are expressed as $\frac{\text{weight of water sorbed}}{\text{weight of solids}} \times 100$. Those obtained by volume contraction were found by extrapolation to the axes of logarithmic curves for volume contraction and degree of sorption and measuring the intercepts as explained previously (3). The cryoscopic values were calculated from the following equation.

$$\text{Grams bound water per 100 grams water in system} = \frac{\Delta_x \times 89.2}{\Delta_s}$$

Where $\Delta$ = Freezing point of sample in solution

$\Delta_a$ = Freezing point after the addition of 32.224 gm. sucrose per 100 gm. water

$\Delta_s = \Delta_a - \Delta$

$\Delta_x = \Delta_s - 2.085$

89.2 = A constant to correct for the hexahydrate formation of sucrose

2.085 = Theoretical freezing point depression caused by sucrose.

The "bound" water corresponding to the degree of sorption is the $\frac{\text{grams "bound" water}}{\text{grams of solids}} \times 100$.

The values for the vapor pressure studies were taken from degree of sorption—vapor pressure curves for the different samples at 80 per cent relative humidity at 20° C. These curves are shown in figure 1.

The shape of the curves makes it apparent that each represents two discontinuous functions. Supplee's results (8) show the same general form,

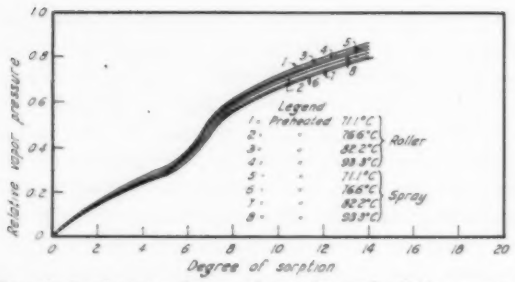


FIG. 1. The relation between degree of sorption and relative vapor pressure in dry milk solids.

while Lampitt and Bushill (19) report that spray-dried milk powder exhibits this feature but atmospheric roll dried milk does not. The shape of the curves results from the fact that the lactose is in the non-crystalline state in the dry material; it takes up water rapidly until sufficiently fluid for orientation of the sugar molecules in a crystalline pattern; then the excess water is free to vaporize and is given up. If the top portion of the curve, which represents water sorption by the proteins, is extrapolated to the point of origin a smooth curve typical of this type of material results.

It will be noted that the curves for all samples are very close together at low water contents and tend to become farther apart as they approach the saturation level.

The comparison of the results by the three methods is shown in table 1.

TABLE 1

*Comparison of results by cryoscopic, vapor pressure, and volume contraction methods for determining the degree of water sorption by dry milk solids*

| Sample preheating temp. (30 min.) | Degree of sorption | | |
|---|---|---|---|
| | Cryoscopic method | Vapor pressure method at 80% rel. humidity | Volume contraction method |
| °C. | *Per cent* | *Per cent* | *Per cent* |
| 71.1 roll | 24.6 | 12.0 | 15.1 |
| 76.6 roll | 26.9 | 13.3 | 18.5 |
| 82.2 roll | 24.9 | 12.4 | 15.8 |
| 93.3 roll | 25.6 | 12.4 | 16.5 |
| 71.1 spray | 42.6 | 12.7 | 29.5 |
| 76.6 spray | 53.8 | 14.2 | 36.3 |
| 82.2 spray | 46.7 | 13.9 | 23.9 |
| 93.3 spray | 44.7 | 12.8 | 23.9 |

Particular attention is called to the fact that the vapor pressure values were taken at 80 per cent relative humidity and so do not indicate the maximum extent to which sorption may occur when exposed to atmospheres saturated with water vapor. The values by the other methods represent maxima for each method.

It will be observed that there is fair qualitative agreement, but that the methods differ in the magnitude of the results. The cryoscopic method gives values greater than either of the others and it seems probable that the vapor pressure method will yield the smallest values although it is difficult to predict the ultimate amount of water sorption that might be obtained if measurements could be made accurately at the saturation level. In all cases the samples preheated at 76.6° C. (170° F.) for 30 minutes showed the greatest water-holding capacity and those prepared by the spray process were superior in this respect to the ones prepared on the atmospheric roll.

## DISCUSSION

It is apparent from the qualitative agreement of the values reported that the methods measure either the same phenomenon or a closely related one. Some possible reasons for the lack of quantitative agreement need to be considered. Gortner (20) has expressed the opinion that the cryoscopic technique may be expected to yield minimal values. He points out that the dissolving of sucrose and subsequent freezing might be expected to shift the "bound" water $\rightleftarrows$ "free" water equilibrium to the right with the result that the values obtained at the freezing point would be lower than those obtained at higher temperatures. Considering the phenomenon from another approach, however, it appears that the equilibrium might well be shifted to the left. The sorption of water is an exothermic reaction and should increase as the temperature is lowered. Data obtained in this laboratory (21) from vapor pressure measurements indicates that dry milk solids will sorb 10.3 gm. water per 100 gm. solids at 20° C. and 14.3 gm. at 10° C. with relative humidity of 70 per cent. The difference tends to increase at higher humidities. Svedberg's data (12) show the same temperature effect. In carrying out the cryoscopic technique, even though the material is allowed to come to equilibrium at 20° C., the temperature must be lowered to the freezing point with a thermal disturbance of the equilibrium. It is perhaps significant that the equilibrium is not disturbed in either the vapor pressure or the volume contraction methods.

It should be mentioned that the vapor pressure method is the only one of the three capable of thermodynamic interpretation involving the calculation of the entropy and free energy changes. This relies on the assumption that the reaction is reversible, which is not strictly true because of the hysteresis effect; and also on the assumption that the system is homogeneous, which again is not true for dry milk solids. For these reasons the thermodynamic calculations are not given.

It is the belief of the author that the results obtained by the volume contraction method are more nearly a correct measure of the degree of sorption than those obtained by the other methods. The volume contraction is a static method and nothing is done to disturb the equilibrium as is the case with the cryoscopic method. Also, the volume contraction method can be

used to measure the degree of sorption of the dry material in direct contact with liquid water, a condition not possible by the vapor pressure method.

## SUMMARY

1. Comparisons are given for the degree of sorption of dry milk solids as determined by the cryoscopic, the vapor pressure, and the volume contraction methods.

2. It is shown that there is qualitative agreement among the methods for differently processed samples, but the values differ in magnitude depending upon the method used.

3. The cryoscopic results for each sample are greater than those by the other methods, and the results by the vapor pressure method at 80 per cent relative humidity are the smallest.

4. Some possible reasons for the differences are discussed.

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# THE FINAL SOLUBILITY OF d-GALACTOSE IN WATER

STEPHEN P. GOULD*

*Division of Dairy Research Laboratories, Bureau of Dairy Industry, U. S. Department of Agriculture*

In the course of some research in this laboratory on sugars, information on the solubility of d-galactose in water was required. The only value found in the literature is that of Dehn (1), who gives the solubility of galactose as 68.30 grams per 100 grams of water at 20–25° C. He does not state whether he used d-galactose nor whether his value refers to a solution in which α–β equilibrium had been attained.

The d-galactose used was a high-grade Pfanstiehl product. Its specific rotation was obtained and was for a 10 per cent solution by volume $[\alpha]_D^{20^\circ} = +80.4°$. Bacteriological tests showed the absence of glucose and lactose.[1] Thus a satisfactory degree of purity was indicated.

Saturated galactose solutions were prepared in two ways, the first by shaking the sugar with water at room temperature, and the second by heating the solution to 40° C. Thus equilibrium was approached from both an undersaturated and a supersaturated condition. Excess crystals were present, of course, in each case. The bottles were suspended in a thermostat kept at 25° C. ± .02. After the solution stood for several days to permit equilibrium among isomeric forms to become established, 5 cc. samples were pipetted from each bottle, run into tared evaporating dishes and weighed. After being heated in a water-jacketed vacuum oven for several days, the dishes were weighed, and the operations repeated until constant weights were obtained. The values for each method of approach to equilibrium were in satisfactory agreement. The mean of these values gave the solubility at 25° C. as 32.09 grams per 100 grams solution. This is equivalent to a solubility of 47.25 grams of galactose in 100 grams of water.

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Received for publication September 1, 1939.

* Deceased Sept. 12, 1939. Any communications regarding this paper should be addressed to E. O. Whittier, of the Bureau of Dairy Industry, who directed the investigation.

[1] These tests were carried out by Dr. H. R. Curren of these laboratories, to whom thanks are due.

# A METHOD FOR CALCULATING THE BAUMÉ READING OF CONDENSED ICE CREAM MIXES*

R. A. LARSON[1] AND P. S. LUCAS

*Dept. of Dairy Husbandry, Michigan State College, East Lansing, Mich.*

The manufacture of ice cream mixes in the vacuum pan has created a need for a method for the accurate forecasting of the Baumé hydrometer reading of these mixes, in order to prevent over or under condensation, and to provide a guide for indicating when the mix is ready to draw. This Baumé reading has been and is still largely determined by taking samples at varying degrees of condensation until the composition of the mix has reached the point desired. Subsequent mixes are then drawn at this predetermined reading. Apparently it should be possible to predict the proper hydrometer readings by paper analysis for mixes of given composition, provided the specific gravities of the ingredients making up the mix are known. When these methods of calculation are checked against actual analyses, however, the relationship shows considerable inaccuracy, sufficiently so as to render their commercial usage impractical.

Sharp and Hart (1) in mentioning 36 formulas for calculation of the relationship between solids and fat content of milk state an important observation, "a large part of this lack of agreement and reproducibility is due to one factor which has never been limited adequately, namely, the lag in the change in the physical state of the fat as the temperature is adjusted to that at which the specific gravity is determined." The difficulties of ascertaining specific gravity mentioned by Sharp and Hart are explained in part by Leighton, Leviton, and Williams (2, 3, 4) in their work on apparent and basic viscosity. These phenomena are variously attributed to fat clumping, specific heat, agitation, acidity, hydration, homogenization, pasteurization, forewarming temperatures, and electrical charges carried.

The Laboratory Manual of the I.A.M.D. (5) carries a table listing the Baumé readings for four mixes of varying compositions taken at five temperatures, varying from 60° F. to 140° F. Sommer (6) gave the following formula for predicting with fair accuracy the specific gravity of a mix being condensed:

$$\text{Specific gravity at } 60^\circ \text{ F.} = \frac{100}{\frac{\%\ \text{Fat}}{.93} + \frac{\%\ \text{Sugar, Serum Solids, and Gelatin}}{1.58} + \%\ \text{Water}}$$

In the same text the specific gravity of milk-solids-not-fat was quoted as 1.5847, of sugar as 1.58, of butterfat as .93, and that of gelatin assumed to be

Received for publication, September 5, 1939.

* Journal Article No. 387 n. s., Michigan State College.

[1] Dairy Industries Supply Association Fellow.

1.58. The temperature at which each determination was made was 60° F. The values given by Sommer are those, he states, of Fleischman.

## EXPERIMENTAL

### *Estimation of Specific Gravity*

Without the interference of such factors as viscosity, surface tension, or specific heat of the various ingredients, the specific gravity of any desired composition ice cream mix could be predicted before condensing the mix, if the specific gravity of each ingredient was known. To illustrate, specific gravity of the following mix could be calculated in this manner:

| | | | | | | |
|---|---|---|---|---|---|---|
| 12. | pounds | butterfat | × | density | = | fat density factor |
| 10. | " | S.N.F. | × | " | = | S.N.F. density fatcor |
| 15. | " | sugar | × | " | = | sugar density factor |
| 0.4 | " | gelatin | × | " | = | gelatin density factor |
| 62.6 | " | water | × | " | = | water density factor |
| 100.0 | pounds | | | | | sum of density factors |

Predicted density = sum of density factors ÷ 100. Since, specific gravity = $\frac{145}{145 - {}^\circ \text{ Baumé}}$, or $^\circ\text{Baumé} = 145 - \frac{145}{\text{specific gravity}}$, such predicted density could be readily converted into degrees Baumé. Application of the above to practical conditions soon proved the fallacy of the reasoning. The specific gravity values given by Fleischman were determined at 60° F., whereas Baumé readings of mixes are usually made at 125° F. The object in this study has been primarily the development of a formula for accurately predicting the specific gravity and Baumé reading of ice cream mixes condensed to a predetermined content. This made necessary determinations of densities of the commoner ingredients used in ice cream, actual condensation of mixes both in laboratory and commercial size vacuum pans, temperature effect and temperature correction on Baumé readings, surface tension, and apparent and basic viscosity values for pan condensed mixes.

### *Condensations in the Laboratory Pan*

A small experimental laboratory type vacuum pan was used for the purpose of preliminary work and with the thought of applying the findings to the operation of the larger factory size pan. It was possible to produce five pounds of finished mix with this apparatus. Four basic mixes were used. These covered in range, so far as commercial practice is concerned, the mixes commonly made in the vacuum pan.

### *Condensations in the Commercial Size Pan*

Mixes made up in the 42 inch vacuum pan were calculated by the normal equation method and were of the varying compositions shown in table 1. The liquid ingredients were heated in the hot well to 95° F., the sugar and

TABLE 1

*Composition of basic mixes used in experiment*

| Mix number | Fat | Solids-not-fat | Sugar | Gelatin |
|---|---|---|---|---|
| | *per cent* | *per cent* | *per cent* | *per cent* |
| 1 | 8 | 11 | 14 | 0.4 |
| 2 | 8 | 11 | 15 | 0.4 |
| 3 | 8 | 11 | 16 | 0.4 |
| 4 | 10 | 10.5 | 14 | 0.4 |
| 5 | 10 | 10.5 | 15 | 0.4 |
| 6 | 10 | 10.5 | 16 | 0.4 |
| 7 | 12 | 10 | 14 | 0.4 |
| 8 | 12 | 10 | 15 | 0.4 |
| 9 | 12 | 10 | 16 | 0.4 |
| 10 | 14 | 9 | 14 | 0.4 |
| 11 | 14 | 9 | 15 | 0.4 |
| 12 | 14 | 9 | 16 | 0.4 |

gelatin added, the mix further heated to 155–160° F. with live steam, drawn into the pan and condensed to the hydrometer readings indicated by the results obtained through the trials with the laboratory size pan. About one hour was required to condense a 1000-pound mix and all mixes were 1000 or 1235 pounds in size. If condensed too far the mixes were standardized with water to desired weight. Samples for viscosity and surface tension determinations were taken after homogenization at 2300 pounds pressure. The mix was then cooled immediately, and tested in the manner described in the preliminary study. Baumé readings were made within an hour after the mixes were drawn. In making these the sample was heated to 155° F., and tested at 5° F. intervals from 155° F. to 60° F. Unless the sample was stirred occasionally in the hydrometer cylinder there was a tendency for it to oil off. Again, as in the preliminary work, pycnometer determinations for density at 70° F. were made for comparison with density determinations at 70° F., the latter being calculated by conversion to density of Baumé hydrometer determinations. For restandardization of mixes Mojonnier tests for fat and total solids were used.

### *Density Determinations of Ice Cream Ingredients*

In an effort to utilize the simple calculation described under "Estimation of Specific Gravity" it seemed desirable to reestimate the specific gravity of the more common ingredients of ice cream and to make this estimation by what might be regarded as an unorthodox method, that of determining the specific gravity of the ingredient in solution or suspension. The specific gravity of each constituent could then be converted to Baumé hydrometer reading using the formula, $^\circ \text{Baumé} = 145 - \frac{145}{\text{specific gravity}}$. Since, in practice, it was learned that the use of the method of calculation mentioned gave inaccurate results, it was hoped that the effect of factors such as condi-

tion of fat, viscosity, and related factors causing the inaccuracy could be compensated for by the determination of a factor that might be used in connection with the calculation.

To make the term specific gravity synonymous with relative density, all weights were determined on an equal arm balance. If the materials in a mix do not expand when heated, density will remain the same. Liquid ice cream mix expands when heated, but so far as could be measured by ordinary means, no solid in the mix, except butterfat, expands under the influence of heat. This factor introduces a slight error in the calculations which follow. The Baumé hydrometer is calibrated to compensate for the approximate errors due to temperature variations, and this correction is probably sufficient for practical purposes. Since coefficients of expansion vary greatly for different liquids, the correction cannot be strictly accurate.

Baumé readings were made of all mixes condensed and the readings converted into density values using the formula previously given. This calculated density was then checked by a density value determined by the pycnometer method, using the mix and water at identical temperatures.

The method adopted for specific gravity determination would seem to eliminate objections to the capsule and displacement methods and to take into consideration the insoluble, colloidally soluble, and wholly soluble constituents of the mix. That porosity of water cannot provide sufficient space for sucrose is shown by the fact that when added to a measured volume of water in quantities less than saturation the volume of the mixture increases.

The method of density determination consisted in the use of a 100 ml. volumetric flask the neck of which was graduated by 0.1 ml. from 100 to 110 ml. Ten grams of skimmilk powder, previously tested for water and fat content, were weighed into the flask, and 100 ml. of boiled distilled water added at 68° F. The mixture was shaken, allowed to stand until all foam had disappeared, and volume determinations made at 5° F. intervals from the temperatures of 60° F. to 155° F. The purpose of this was to eliminate necessity of correction for expansion of glass. Several duplicate determinations were made, as well as determinations using 12 and 14 grams of skimmilk powder per 100 grams of water. The increase in the volume of water in the flask was recorded as the volume of the particular weight of skimmilk powder added.

The above determination was checked for accuracy of volume at different temperatures by calculation of density thru the use of a 25 ml. pycnometer. Weighings were made into the pycnometer at room temperature, the filled pycnometer heated to the desired temperature, dried, reweighed, and the density of its mixture content calculated by determination of the pycnometer volume at different temperatures and dividing this volume into the pycnometer weight of the mixture. By determining volume of water and mixture at the different temperatures used, no correction for glass expan-

sion was necessary. The density of the mixture was taken as the weight of all water present plus weight of dry powder divided by the volume of all water present plus the volume of dry powder. The density of the solids-not-fat was taken as the weight of dry powder divided by the volume of dry powder. Although in the same condition as when weighed into the ice cream mix the density values are not strictly accurate, due to water of hydration. A second factor causing slight inaccuracy was the fat content of the skimmilk powder, amounting to 0.1 per cent. This, calculated as water, caused a difference of less than 0.0001 in the density of solids-not-fat.

The values for density of water at varying temperatures given in Lange's Handbook of Chemistry (7) and for the density of butterfat as given by Bailey (8) were used for this work.

### *Predicted Density Determinations*

Once the densities of the various ice cream constituents were available an attempt was made to use the values for prediction of correct Baumé readings at which to draw mixes from the vacuum pan. This was done by comparing at the same temperature predicted Baumé, as calculated from density, with the Baumé reading of a mix previously made of known composition. If the two were not the same, the factor by which the predicted density should be multiplied to give a correct reading, was determined. Should this be possible it would indicate existence of a straight line relationship between the additive density of a mix and its measured density. For this reason additive densities were calculated to the fourth decimal place and the Baumé hydrometer read as nearly as possible to its smallest division, 0.1°.

## RESULTS

### *Condensation in Laboratory and Commercial Pans*

*Physical state of fat, effect on Baumé reading.* When mixes stand or cool the increase in viscosity will cause the hydrometer reading to vary, due not only to viscosity but probably due also to mechanical resistance offered by solidified and crystallized butterfat.

TABLE 2

*Average effect of state of fat on Baumé reading of ice cream mixes*

| Composition of mix | Baumé reading with fat in liquid state | Baumé reading with fat in solid state |
|---|---|---|
| 8: 11 : 14: 0.4 | 13.10 | 13.25 |
| 8: 11 : 15: 0.4 | 13.35 | 13.50 |
| 8: 11 : 16: 0.4 | 13.75 | 13.90 |
| 10: 10.5: 14: 0.4 | 12.30 | 12.50 |
| 10: 10.5: 14: 0.4 | 12.80 | 12.95 |
| 10: 10.5: 14: 0.4 | 13.30 | 13.45 |
| 12: 10 : 14: 0.4 | 11.80 | 12.00 |
| 14: 9 : 14: 0.4 | 11.20 | 11.40 |

The samples above were cooled to 60° F., held four hours at 40° F., warmed to 60° F., and Baumé determinations made. Duplicates of these samples were cooled to 60° F., held four hours at 40° F., heated to 155° F., cooled to 60° F. and tested for density. These duplicates, it will be noted from the second column of the table, gave lower readings, doubtlessly, because the fat was still in an uncrystallized state. The results would indicate the necessity of treating cold samples, as indicated by the treatment given the duplicates, to secure a correct Baumé reading.

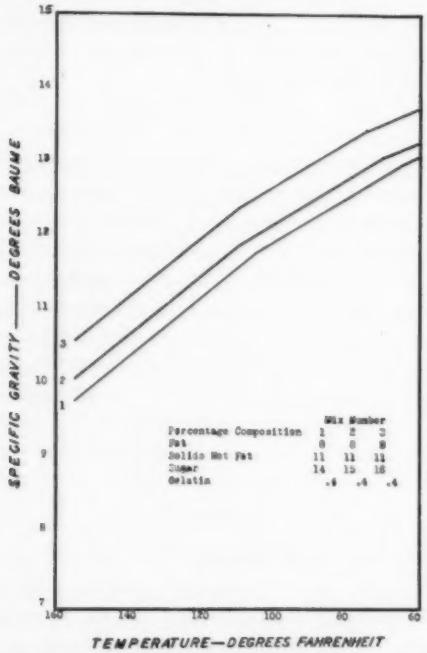


FIG. 1. Relation between temperature and Baumé readings of different composition ice cream mixes.

*Temperature effect on Baumé readings.* The Baumé readings were made from 155° F. to 60° F. at 5° F. intervals in the hope of obtaining an ideal temperature at which to take readings.

Within the range of 110° F. to 155° F., each 5° F. interval caused a change of 0.2° in the Baumé reading; from 100° F. to 70° F. the change in Baumé was 0.15° for each 5° F. change; and for 60° F. to 65° F., in most cases, 0.1° Baumé change for the 5° F. interval. Lack of uniformity in these changes per 5° F. interval may be due to the changes beginning to take place in the fat globule structure: the change takes place at 110° F.

and this point is fairly close to the solidifying point of butterfat. The time required for butterfat to crystallize would tend, however, to weaken such a theory. It is more likely due to viscosity changes. Inspection of figures 1 to 4 shows composition of mix to have little if any effect.

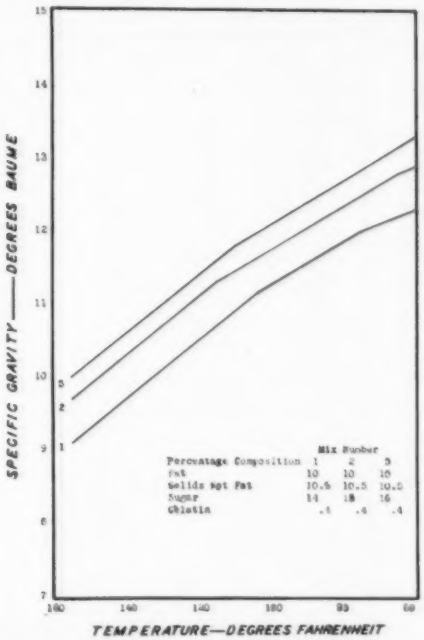
SPECIFIC GRAVITY —— DEGREES BAUME
TEMPERATURE—DEGREES FAHRENHEIT
Mix Number
Percentage Composition 1 2 3
Fat 10 10 10
Solids not Fat 10.5 10.5 10.5
Sugar 14 15 16
Gelatin .4 .4 .4

FIG. 2. Relation between temperature and Baumé readings of different composition ice cream mixes.

*Baumé and pycnometer density determinations compared.* In the following table densities of mixes are given as determined by pycnometer and hydrometer. Each determination given represents the average for each group, composed of at least two, and, in most cases three, individual mixes. Temperature corrections were made to 70° F. Baumé readings were converted to density. Since the pycnometer was calibrated to be used at 68° F. the Baumé hydrometer was corrected to 70° F. to make the results comparable. Compositions were determined by the Mojonnier method.

The above table of results possesses value chiefly as a check on the accuracy of the hydrometer reading, and on the degree of precision with which it may be read. On the average the Baumé converted readings varied 0.0011 from the pycnometer values. This is slightly more than 0.1° Baumé. Nearly one-half of the readings were more than 0.1° Baumé

greater or less than the pycnometer determination. The source of error, therefore, appears to be with hydrometer accuracy and with the operator. The greatest source of mechanical error appears to be with the adjustment of temperature. Since it seems to be possible to read the hydrometer no more closely than 0.1°, and since this variation causes a density variation

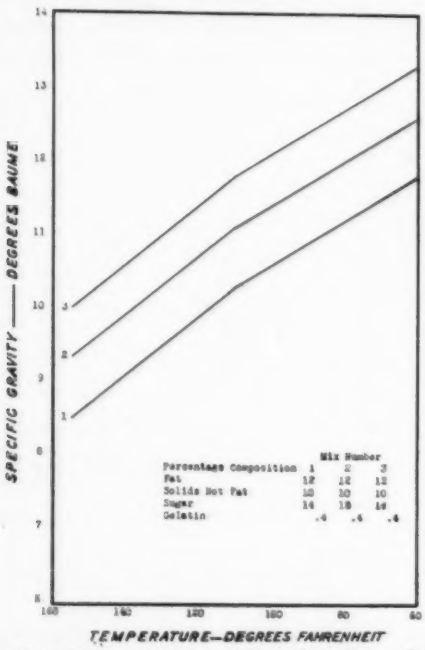
SPECIFIC GRAVITY —— DEGREES BAUMÉ
TEMPERATURE—DEGREES FAHRENHEIT
Mix Number
Percentage Composition 1 2 3
Fat 12 12 12
Solids Not Fat 10 10 10
Sugar 14 18 18
Gelatin .4 .4 .4

FIG. 3. Relation between temperature and Baumé readings of different composition ice cream mixes.

of 0.0008, it would seem advisable that only the best grade of rechecked hydrometers, having easily read graduations, be used.

*Homogenization effect on viscosity and surface tension.* Viscosity and surface tension will undoubtedly affect density as determined by hydrometer and any processing of the mix which greatly affects viscosity and surface tension will cause readings of one mix so treated to appear at variance with an identical mix processed normally. Unfortunately no mixes in this series were made with butter as a source of fat and the results apply only to mixes carrying fat as it occurs in milk and cream. Probably a butter mix would show less viscosity due to dispersion of fat. Viscosity was determined by the Mojonnier-Doolittle and MacMichael viscosimeters; surface tension with a du Nuoy tensiometer.

Unhomogenized mixes made in the laboratory size pan developed con-

TABLE 3

*Average densities at 70° F. of ice cream mixes calculated by Baumé and pycnometer methods*

| Mix | | | | Density | | |
|---|---|---|---|---|---|---|
| Composition Percentage | | | | | | |
| Fat | Solids-not-fat | Sugar | Gelatin | Baumé | Pycnometer | Difference |
| 8 | 11 | 14 | 0.4 | 1.0972 | 1.0980 | 0.0008 |
| 8 | 11 | 15 | 0.4 | 1.1009 | 1.1015 | 0.0006 |
| 8 | 11 | 16 | 0.4 | 1.1032 | 1.1150 | 0.0018 |
| 10 | 10.5 | 14 | 0.4 | 1.0841 | 1.0848 | 0.0007 |
| 10 | 10.5 | 15 | 0.4 | 1.0939 | 1.0934 | 0.0005 |
| 10 | 10.5 | 16 | 0.4 | 1.0985 | 1.1003 | 0.0018 |
| 12 | 10 | 14 | 0.4 | 1.0865 | 1.0890 | 0.0005 |
| 12 | 10 | 15 | 0.4 | 1.0927 | 1.0935 | 0.0008 |
| 12 | 10 | 16 | 0.4 | 1.0985 | 1.1004 | 0.0019 |
| 14 | 9 | 14 | 0.4 | 1.0813 | 1.0825 | 0.0012 |
| 14 | 9 | 15 | 0.4 | 1.0861 | 1.0864 | 0.0003 |
| 14 | 9 | 16 | 0.4 | 1.0927 | 1.0923 | 0.0004 |
| 13.90 | 9.45 | 15 | 0.4 | 1.0856 | 1.0878 | 0.0022 |
| 13.85 | 9.55 | 15 | 0.4 | 1.0886 | 1.0892 | 0.0006 |
| 14.50 | 11.0 | 14 | 0.4 | 1.0877 | 1.0863 | 0.0014 |
| 9.9 | 10.2 | 14 | 0.4 | 1.0841 | 1.0862 | 0.0021 |

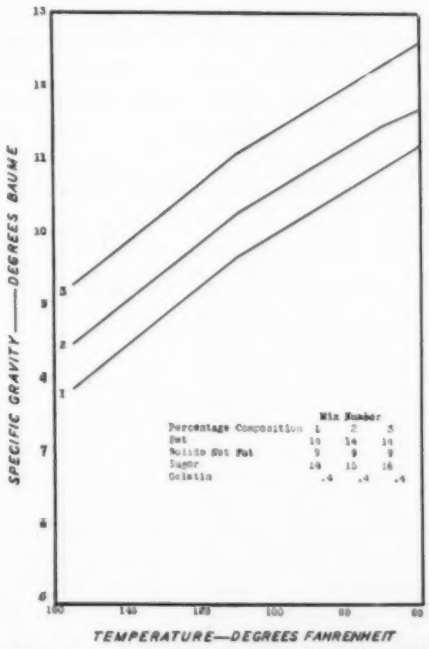


FIG. 4. Relation between temperature and Baumé readings of different composition ice cream mixes.

siderable viscosity, this amounting from one-half to nearly the same viscosity developed by identical homogenized mixes. Homogenization had little effect on viscosity of factory-size pan condensed mixes.

As a general rule increased viscosity was accompanied by a slight lowering of surface tension although the change was not sharp. With the factory-size pan homogenization caused no surface tension changes, although this probably would vary according to pressure and to condition of the homogenizing valve surfaces.

Apparent viscosity varied from twice to three and one-half times the basic viscosity. Pan condensed, homogenized mixes approximately tripled their viscosity during a 24-hour aging period. This tendency was greater with high fat and high total solids mixes. Viscosities measured by the MacMichael viscosimeter in centipoises were about three times as great as the value in degrees retardation secured by the Mojonnier-Doolittle viscosimeter.

### *Density Determination of Ice Cream Ingredients*

*Density of solids-not-fat.* Rather than use data on coefficients of expansion of water, the 110 ml. volumetric flasks used for density determination of the solid ingredients of the mix were calibrated at several temperatures. Holding 100 ml. at 68° F., the same weight of water at 120° F. had risen in the graduated neck to a volume of 101.05 ml. Readings at other temperatures, as recorded below, were secured similarly.

TABLE 4

*Volume readings secured in calibration at varying temperatures of 110 ml. graduated volumetric flasks using boiled, distilled water*

| Temperature degrees Fahr. | Volume in ml. | |
|---|---|---|
| | Flask 1 | Flask 2 |
| 155 | 102.05 | 102.05 |
| 150 | 101.90 | 101.90 |
| 145 | 101.70 | 101.70 |
| 140 | 101.60 | 101.60 |
| 135 | 101.40 | 101.40 |
| 130 | 101.30 | 101.30 |
| 125 | 101.20 | 101.20 |
| 120 | 101.05 | 101.05 |
| 68 | 100.00 | 100.00 |
| 60 | 99.90 | 99.90 |

Increase in temperature and volume of water did not show a straight line relationship. In general the increase in volume was about 0.15 ml. per 5° F. increase in temperature. The values given in table 4 were used for determining the volume per unit of weight of skimmilk powder, gelatin, and sugar in the following manner. Ten grams of skimmilk powder were added to one of the flasks containing 100 ml. of water at 68° F., the temperature

raised to 130° F. and the volume of the mixture taken. This volume of mixture, less 101.3 ml., the volume of the water at 130° F., was taken as the volume of water displaced, or volume occupied by the 10 grams of skimmilk powder when present in water in its three states, solution, colloidal solution, and suspension. Volume of powder divided by weight of skimmilk powder was taken as the density of the skimmilk powder. Corrections were made for moisture and fat content. Densities of gelatin and sucrose were similarly determined.

TABLE 5

*Volume readings of skim milk powder at various temperatures*

| Temp. deg. Fahr. | Volume 100 ml. water | Volume 100 ml. water 10 grams pwd. | Volume 100 ml. water 12 grams pwd. | Volume due to | | Density of S.N.F. |
|---|---|---|---|---|---|---|
| | | | | 10 gms. powder | 12 gms. powder | |
| | ml. | ml. | ml. | ml. | ml. | |
| 60 | 99.90 | 106.10 | 107.34 | 6.30 | 7.44 | 1.6129 |
| 68 | 100.00 | 106.20 | 107.44 | 6.30 | 7.44 | 1.6129 |
| 120 | 101.05 | 107.25 | 108.49 | 6.30 | 7.44 | 1.6129 |
| 125 | 101.20 | 107.40 | 108.64 | 6.30 | 7.44 | 1.6129 |
| 130 | 101.30 | 107.50 | 108.74 | 6.30 | 7.44 | 1.6129 |
| 135 | 101.40 | 107.60 | 108.84 | 6.30 | 7.44 | 1.6129 |
| 140 | 101.60 | 107.80 | 109.04 | 6.30 | 7.44 | 1.6129 |
| 145 | 101.70 | 107.90 | 109.14 | 6.30 | 7.44 | 1.6129 |
| 150 | 101.90 | 108.10 | 109.34 | 6.30 | 7.44 | 1.6129 |
| 155 | 102.05 | 108.25 | 109.49 | 6.30 | 7.44 | 1.6129 |

From table 5, it will be noted that an increase in temperature did not cause a change in volume of the skimmilk powder, using either 10 grams or 12 grams in 100 ml. water. This verified preliminary data secured when 10, 12, and 14 grams of skimmilk powder were used, results of which are not recorded here.

TABLE 6

*Density of solids-not-fat suspensions from volume readings using 10 grams powder*

| Temperature | Density of suspension to 60° F. (10 gms. powder—100 ml. water) | |
|---|---|---|
| | By pycnometer | By volume |
| 60 | 1.0351 | 1.0358 |
| 68 | 1.0351 | 1.0350 |
| 120 | 1.0375 | 1.0355 |
| 125 | 1.0372 | 1.0355 |
| 130 | 1.0364 | 1.0356 |
| 135 | 1.0378 | 1.0358 |
| 140 | 1.0380 | 1.0358 |
| 145 | 1.0367 | 1.0358 |
| 150 | 1.0360 | 1.0359 |
| 155 | 1.0354 | 1.0360 |

Density of SNF at any temperature $= \frac{\text{Wt. SNF}}{\text{Vol. SNF}}$ at any temperature.

Column 2 of table 6 contains the values obtained by pycnometer weights. In each case all determinations were corrected to 60° F., using the values given in table 7, for the reason that the pycnometer was also corrected to 60° F. These were of service in checking individual determinations. The same system was followed for the values in column 3, and for density of solids-not-fat given in table 5. It was followed, also, in the calculations for densities of solids-not-fat, sugar, and gelatin. These were 1.6129, 1.6107, and 1.5384 respectively.

Density calculations gave identical values for solids-not-fat when either 10 or 12 grams of skimmilk powder were used. This is to be expected, however, since the skimmilk solids did not change volume with the variations in heat used. The density value secured, however, was from the suspension and solution of the skimmilk in water, and is literally the density due to the solids-not-fat in suspension in the water. Density of the suspension by the volume and pycnometer methods checked fairly closely.

Density of sugar was the same at all temperatures used. One and two grams of gelatin were used per 100 ml. of water in checking its density. While this is much greater than the amount used in ice cream mix this quantity was necessary for accuracy in the particular method used. As with sugar and solids-not-fat, gelatin did not increase in volume as temperature increased. Its density, therefore, was found to be uniform at all temperatures, within the range used.

*Density of butterfat and water.* The densities of butterfat and water, as given in the following table, are calculated from values given by Bailey and Lange's Handbook of Chemistry previously cited. Bailey gives the density change for butterfat as 0.00038 per degree Fahrenheit change, and the density as 0.9 at 113° F.

TABLE 7

*Density of butterfat and water at various temperatures*

| Temperature deg. Fahr. | Density of butterfat | Density of water |
|---|---|---|
| 60 | 0.92014 | 0.99905 |
| 68 | 0.9016 | 0.99823 |
| 120 | 0.8974 | 0.98856 |
| 125 | 0.8955 | 0.98729 |
| 130 | 0.8936 | 0.98597 |
| 135 | 0.8917 | 0.98507 |
| 140 | 0.8898 | 0.98324 |
| 145 | 0.8879 | 0.98262 |
| 150 | 0.8860 | 0.98032 |
| 155 | 0.8841 | 0.97881 |

Density values above unity are given in the Handbook cited above and the first two columns of table 8 are copied from it. From values given in these two columns, values given in column three were calculated for use in prediction of Baumé reading. Most density readings, covering the normal

range of 1.06 to 1.11, may easily be translated into Baumé reading through the use of the table.

TABLE 8

*Relation between density and Baumé scale for densities above unity*

| Density | Baumé | Density to make 1° Baumé change |
|---|---|---|
| 1.05 | 6.91 | 0.00752 |
| 1.06 | 8.21 | 0.00763 |
| 1.07 | 9.49 | 0.00781 |
| 1.08 | 10.78 | 0.00775 |
| 1.09 | 11.97 | 0.00840 |
| 1.10 | 13.18 | 0.00826 |
| 1.11 | 14.37 | 0.00840 |
| 1.12 | 15.54 | 0.00854 |
| 1.13 | 16.68 | 0.00877 |
| 1.14 | 17.81 | 0.00885 |

*Prediction of Baumé reading according to mix composition.* Inspection of table 8 will show that a change of .0008 in density is necessary to cause a change of 0.1° Baumé. The Baumé hydrometer, under practical conditions, can be read no more closely than 0.1°. It would be possible, therefore, to predict density within 0.0008 provided the hydrometer were read as closely as 0.1°. In preparing composition comparisons of actual determinations by Baumé and calculated Baumé the density values of the ingredients used were, butterfat at 113° F., 0.9; solids-not-fat, 1.613; sugar, 1.61; gelatin, 1.54; and water at 60° F., 0.99823. The densities of the second, third, and fourth were calculated as being constant through the 60–155° F. temperature range. Changes of densities for butterfat and water are given in table 7. Mixes were condensed to a predetermined composition and Baumé readings made at the temperatures specified. These readings were converted to density values. The additive densities at these varying temperatures were then calculated and divided into the corresponding density obtained from the Baumé reading. The result was the factor used for multiplying additive density at the three temperature ranges used. This value times *added density of mix ingredients* gave *factored density* or the density nearest that obtained by actually making and testing the mix. The calculation involved merely the determination of a factor which, when multiplied by predicted density, gave a value that could be converted into the proper Baumé at which the mix was to be drawn.

Seventy-two mixes were made up to desired composition and their Baumé test read. The additive density of these mixes was calculated for the temperature range of 120–135° F. When multiplied by the factor 0.949 the average difference for the 72 mixes between predicted and determined density was 0.00175. This density variation would amount to about a 0.2° variation in Baumé, within the range specified, and, normally, all commercially condensed mixes are read within this range.

With the mix at 60° F. at which it is more viscous and the fat in a solid state, and using the factor 0.955, an average accuracy of 0.2° Baumé was obtained with the 18 readings observed. When Baumé reading was calculated from additive density the extreme variation from the observed reading was 0.7°. Of the 18 readings at 60° F. the predicted density was within a reading range of 0.1° Baumé of observed readings in 33 per cent of the readings. This number of checks, using the factor 0.955, is perhaps too few to be considered conclusive.

Using the factor 0.949 to predict the density of a mix within the temperature range of 120–135° F., 45.83 per cent of the readings were within 0.1° of the observed Baumé reading. Of the 90 readings taken, 45.55 per cent were within 0.1° Baumé of observed readings. Forty-three readings were higher than 0.0016 density allowance for 0.2° Baumé and 47 readings were below this value.

*Sources of Error*

The use of these factors for the temperature ranges mentioned was necessary because of the conditions which tended to change the direction of the lines showing the relationship between specific gravity and temperature (figures 1 to 4 inclusive). There may be other sources of error, also, in predicting density, most of which are directly related to accuracy in making readings and tests. Thus a density variation of 0.0008 corresponds to 0.1° Baumé change. A slight error in the Mojonnier determination for fat or total solids would easily cause 0.1° Baumé reading change. In calculating the mix, an accurate determination of fat and solids must be made rather than fat determination only, with assumption that solids-not-fat are present to the extent of nine per cent. An error in this determination may be as important as an error in the Baumé reading itself or one in predicting density. This is true especially at the higher temperature ranges where the differences between the density of water and butterfat, and those of sugar, milk-solids-not-fat, and gelatin are marked.

Losses of portions of the mix ingredients, especially solids, during condensing will alter the mix composition. When the product is standardized back these are not accounted for. The composition, therefore, is not exactly as calculated, and predicted densities from composition are not strictly correct. An error of 0.1 per cent in solids-not-fat test causes an apparent increase or decrease of 0.1 per cent water, causing a change of 0.0008 in density or 0.1° Baumé reading, *i.e.*, when the reading is made at 125° F., which is normally the temperature at which it is made, and with a mix of 8 : 11 : 14 : 0.4 composition. Slight changing of the amounts of those solids heavier than water would cause a corresponding change in Baumé reading.

Using specially built apparatus having greater accuracy in measurement of volume there is the possibility that additive densities at different temperatures would not give a graph in the form of a straight line. Using the

apparatus described in this study, however, the changes in volume, if any, were too small to be observed when the material was in suspension and solution in water. With volumetric apparatus reading to fractional parts of a milliliter it is believed that very exact Baumé determinations could be predicted from desired composition of mix.

## SUMMARY

The Baumé hydrometer may be used as an accurate indicator of the composition of ice cream mixes during condensation and as a method for prediction of when to strike the batch. This work was designed to originate a method for prediction of Baumé readings for mixes of a definite finished composition, using composition as the basis for calculation. In preliminary work, homogenization of the mix was found to have no effect on Baumé reading; that, to be comparable from day to day, readings had to be taken at the same temperature; that the average mix condensed at a 24 inch vacuum boiled at 140° F., which made the temperature of 125° F. a convenient one at which to take Baumé readings; that density of all mixes studied varied 0.0016 and Baumé reading varied 0.2° for each 5° F. change in temperature within the range of 115–155° F.; that pan condensed mixes are similar in viscosity and surface tension to vat prepared mixes; and that, as a rule, great increase in viscosity resulted in slight increase in surface tension.

Twelve mixes of different compositions were made up, condensed, analyzed, restandardized to desired composition and their Baumé readings taken for use as standards. By using this standard it was found that a 1235 pound mix could be drawn to within ten pounds of its desired weight. Within the range of 60° F. to 155° F. milk solids-not-fat, sugar, and gelatin were found with increase in temperature to remain constant in volume when in suspension or solution. Densities of ice cream ingredients, determined by the methods described, were found to be as follows: milk-solids-not-fat, 1.6129; sucrose, 1.6107; and gelatin, 1.5384. Use was made of densities of water and butterfat, as given in table 7.

Additive density was calculated, as described, using the value for water and butterfat at the temperature at which the Baumé was to be read. This additive density, divided by 100, and the result multiplied by the factor, 0.949, for readings within the temperature range of 120° F. to 135° F., gave a predicted density, which when converted to Baumé give an average density accuracy within 0.2° Baumé.

To make use of these values in predicting Baumé reading for a mix the number of pounds of each component of the mix in 100 pounds is multiplied by the density of the ingredient at the temperature at which the Baumé reading is to be made. Density of milk-solids-not-fat, sugar, gelatin, and butterfat and water have been given. The density factors are added, di-

vided by 100, and multiplied by 0.949, if the Baumé reading is to be made between the optimum temperature range of 120° F. to 135° F. The density value secured is converted to Baumé by reference to table 8. This value should give correct Baumé, within 0.2°, at which to strike the mix.

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# SOME FACTORS RESPONSIBLE FOR VARIATIONS IN THE ACID NUMBERS OF THE FAT IN CREAM AND IN COMMERCIAL BUTTER[1]

E. L. FOUTS

*Dairy Department, Oklahoma A. and M. College, Stillwater, Oklahoma*

In a previous article it was shown that the acid numbers of the fat of commercial butter varied rather widely. No close correlation existed between the acid number of the fat and the quality of commercial unsalted butter; butter of good quality often had relatively high acid numbers, while some rancid samples had relatively low acid numbers. In order to determine some of the possible causes for the significant fat acid numbers variations observed in commercial butter a number of experimental trials were made.

## EFFECT OF THE NORMAL MIXED FLORA AND MILK LIPASE IN RAW CREAM ON THE ACID NUMBER OF THE FAT

Raw cream from several sources was used in the trials to determine the degree of hydrolysis caused by lipolytic organisms and by lipase in cream. Palmer (12) reported that 1 part of formaldehyde in 1500 parts of cream inhibited the growth of most organisms with no deterimental effect on the milk lipase.[2] In order to determine the effect of formaldehyde on pure cultures of some of the lipolytic organisms commonly present in raw cream, small lots of sterilized cream were inoculated with known lipolytic organisms. Formaldehyde was immediately added to the cream in concentrations ranging from 1 part in 4800 parts to 1 part in 1200 parts of cream. These lots of cream were churned after holding 6 days at 21° C. The effect of formaldehyde on the organisms was determined by their ability to grow as evidenced by increases in the acid number of the fat.

Organisms varied considerably in their tolerance for formaldehyde (table 1). *Ach. lipolyticum, Myc. lipolytica* and *Ps. fluorescens* grew very little in a concentration of 1 part formaldehyde to 4800 parts of cream, while *O. lactis* grew luxuriantly in all concentrations up to 1 part in 2000 parts of cream. None of the organisms showed appreciable activity in cream containing 1 part of formaldehyde in 1600 parts of cream. As a result of these trials it was assumed that any lipolysis which occurred in raw cream containing 1 part formaldehyde to 1500 parts cream was due largely to the action of

Received for publication September 11, 1939.

[1] From a portion of a thesis presented to the Graduate School of Iowa State College in partial fulfillment of the requirements for the Ph.D. degree.

[2] Recent work by B. L. Herrington and V. N. Krukovsky has established the presence of two lipases in milk. One is inhibited completely by small amounts of formaldehyde, the other is not sensitive to moderate amounts of it. J. DAIRY SC., 22: 127–135. 1939.

milk lipase while in the cream containing no formaldehyde, the lipolysis was due to the combined action of lipase and micro-organisms.

TABLE 1

*Resistance of certain lipolytic microorganisms to formaldehyde in cream (Acid number of original fat 0.6)*

| Concentration of formaldehyde in cream | Acid number of butterfat after incubating cream 6 days at 21° C. | | | |
|---|---|---|---|---|
| | Cream inoculated with | | | |
| | *Ach. lipolyticum* | *Myc. lipolytica* | *Ps. fluorescens* | *O. lactis* |
| 0 | 3.3 | 30.6 | 5.0 | 16.3 |
| 1–4800 | 1.0 | 1.3 | 1.5 | 16.4 |
| 1–3600 | .9 | 1.8 | 1.4 | 13.3 |
| 1–2400 | .9 | 1.2 | 1.2 | 7.9 |
| 1–2000 | .9 | 1.1 | 1.1 | 6.7 |
| 1–1600 | .9 | 1.2 | 1.0 | .8 |
| 1–1400 | .9 | 1.3 | 1.0 | .7 |
| 1–1200 | 1.0 | 1.5 | 1.2 | .8 |

Small portions of several lots of raw cream from different sources, with and without formaldehyde added, were stored for 2, 4, 6, 10 and 14 days at 5°, 13° and 21° C. The lots of cream were then churned and acid numbers of the butterfat determined.

The data shown in tables 2 and 3 reveal that in general, the lipolysis in the samples containing no formaldehyde was greatest at the lowest temperature. At 21° C. the rate of acid formation was considerably greater than at the lower temperatures which apparently tended to check the growth of some of the lipolytic organisms. Exceptions to this generalization were occasionally encountered, as for example in table 2, the cream stored at 21° C. for 14 days showed a higher fat acid number than did another lot of the same cream stored at lower temperatures. In the samples containing formaldehyde supposedly only the milk lipase was active in splitting the fat. The lipase in these samples caused hydrolysis at all temperatures studied but increases in its activity were evident as the temperatures of storage increased. The acid numbers of the fat of one group of these samples containing formaldehyde (table 2) after 14 days storage at 5°, 13° and 21° C. were 6.0, 7.5 and 13.2, respectively. Increases in the titratable acidity of the samples of cream treated with formaldehyde also occurred at all temperatures studied. From the same table it may be observed that from the original titratable acidity of 0.16 per cent, the acidity of the samples containing formaldehyde increased in 14 days at 5°, 13° and 21° C. to 0.36, 0.40 and 0.48 per cent, respectively. These increases were probably due to liberation of certain fatty acids by lipase. Increases in the acid numbers of the fat were roughly proportional to the increases in titratable acidity. The increases in acid numbers and titratable acidities were thought to be

TABLE 2

*Effect of the normal mixed flora and milk lipase in raw cream on the acid number of the fat*
*(Cream separated from mixed milk of several cows)*

| Days held at | Normal raw cream | | | Formaldehyde added (1–1500) | | |
|---|---|---|---|---|---|---|
| | Per cent acidity | Acid number | Flavor | Per cent acidity | Acid number | Flavor |
| *5° C.* | | | | | | |
| 0 | 0.16 | 0.9 | good | 0.16 | 0.9 | good* |
| 2 | .26 | 6.5 | bitter, rancid | .21 | 3.5 | oxidized |
| 4 | .35 | 13.2 | bitter, rancid | .24 | 3.9 | oxidized |
| 6 | .51 | 11.1 | bitter, rancid | .28 | 4.5 | rancid |
| 10 | .60 | 15.2 | bitter, rancid | .28 | 5.3 | rancid |
| 14 | .69 | 17.1 | bitter, rancid | .36 | 6.0 | rancid |
| *13° C.* | | | | | | |
| 0 | .16 | .9 | good | .16 | .9 | good |
| 2 | .58 | 7.6 | rancid | .22 | 4.0 | rancid |
| 4 | .70 | 8.2 | rancid | .24 | 4.8 | rancid |
| 6 | .88 | 7.6 | rancid | .30 | 5.5 | rancid |
| 10 | 1.00 | 10.1 | rancid | .35 | 6.2 | rancid |
| 14 | 1.14 | 11.5 | rancid | .40 | 7.5 | rancid |
| *21° C.* | | | | | | |
| 0 | .16 | .9 | good | .16 | .9 | good |
| 2 | .70 | 6.5 | good | .22 | 4.9 | oxidized |
| 4 | .70 | 8.2 | very sour | .28 | 4.8 | oxidized |
| 6 | .97 | 9.0 | rancid, cheesy | .38 | 7.5 | rancid |
| 10 | 1.03 | 11.5 | rancid, cheesy | .40 | 9.5 | rancid |
| 14 | 1.05 | 22.2 | rancid, cheesy | .48 | 13.2 | rancid |

* Formaldehyde was detectable in all samples containing it; the term "good" was used to indicate the absence of a rancid flavor or odor.

due largely to the action of milk lipase on the fat since plate counts on these samples revealed relatively few organisms. Rarely did the plates show more than a few hundred organisms per milliliter. Long (10) and Collins (3) reported that organisms must be present in reasonably large numbers to cause defects and it is believed that there were too few organisms in these samples to cause the acidity increases observed. This statement agrees with the findings of Krukovsky and Sharp (8) who showed that raw milk on standing at temperatures too low for bacterial growth, increased considerably in titratable acidity.

In some of the samples containing no formaldehyde marked increases in the acid number of the fat were noted between 10 and 14 days of storage. These sharp increases probably were caused by more rapid mold growth during this period.

The data in table 4 show that when milk from individual cows was held at 5° C., fatty decomposition occurred due to growth of organisms and action of lipase very similar to that observed in mixed herd milk.

Of the two biological agencies capable of causing lipolysis as measured by the acid number of the fat, the action of micro-organisms was of somewhat greater importance than the action of milk lipase. In the raw cream

TABLE 3

*Effect of the normal mixed flora and milk lipase in raw cream on the acid number of the fat*
*(Cream separated from mixed milk of several cows)*

| Days held at | Normal raw cream | | | Formaldehyde added (1–1500) | | |
|---|---|---|---|---|---|---|
| | Per cent acidity | Acid number | Flavor | Per cent acidity | Acid number | Flavor |
| *5° C.* | | | | | | |
| 0 | 0.14 | 0.6 | good | 0.14 | 0.6 | good* |
| 2 | .26 | 6.5 | rancid | .21 | 3.5 | rancid |
| 4 | .35 | 13.2 | rancid | .24 | 3.6 | rancid |
| 6 | .51 | 16.0 | rancid | .28 | 4.1 | rancid |
| 10 | .62 | 21.1 | rancid | .30 | 5.0 | rancid |
| 14 | .75 | 27.0 | rancid | .33 | 6.8 | rancid |
| *13° C.* | | | | | | |
| 0 | .14 | .6 | good | .14 | .6 | good |
| 2 | .58 | 7.6 | rancid | .22 | 4.0 | rancid |
| 4 | .70 | 8.4 | rancid | .24 | 4.8 | rancid |
| 6 | .88 | 9.1 | rancid | .30 | 5.0 | rancid |
| 10 | .90 | 11.2 | rancid | .32 | 6.3 | rancid |
| 14 | .92 | 13.1 | rancid | .34 | 7.5 | rancid |
| *21° C.* | | | | | | |
| 0 | .14 | .6 | good | .14 | .6 | good |
| 2 | .70 | 6.5 | rancid | .22 | 4.9 | rancid |
| 4 | .76 | 9.6 | rancid | .28 | 5.7 | rancid |
| 6 | .96 | 11.0 | rancid | .38 | 6.0 | rancid |
| 10 | .95 | 12.3 | rancid | .40 | 7.5 | rancid |
| 14 | .97 | 14.5 | rancid | .41 | 9.2 | rancid |

* See footnote, table 2.

in which both micro-organisms and normal milk lipase had been active the acid numbers of the fat were often 3 to 4 times greater than in the samples containing formaldehyde. This is particularly well demonstrated by the data shown in table 3. The samples held at 5° C. containing formaldehyde at 4, 6, 10 and 14 days had acid numbers of 3.6, 4.1, 5.0 and 6.8, respectively while similarly held samples of the same cream containing no formaldehyde had acid numbers of 13.2, 16.0, 21.1 and 27.0, respectively. The increases in acid numbers due to growth of micro-organisms were much greater than the increases due to milk lipase. The data presented reveal an unusual circumstance in that in every case the total lipolysis at 13° C. was less than at either 5° or 21° C. after 14 days of storage. No explanation is offered for this condition.

Davies (4) reported that certain metals tended to inhibit lipase activity in butter. In order of their inhibiting power were copper, iron, nickel, cobalt, manganese and chromium. Tin and aluminum had no effect. In the trials reported no effort was made to check or control the normal metal contamination. The results obtained may have been influenced by this factor.

Rice and Markley (13) reported that one of the causes of rancidity in dairy products is the carrying over of the enzyme into the manufactured

products. In view of the wide use of the pasteurization process for dairy products it seems doubtful that a significant carry-over of lipase would occur under ordinary factory conditions.

TABLE 4

*Effect of the normal mixed flora and milk lipase in raw cream on the acid number of the fat*
*(Cream separated from milk of individual cows)*

| Cow | Fresh cream | | Cream after 14 days at 5° C. | | | |
|---|---|---|---|---|---|---|
| | | | Normal | | Formaldehyde added (1–1500) | |
| | Acid number of fat | Flavor | Acid number | Flavor | Acid number | Flavor |
| 1 | 0.5 | good | 4.0 | rancid, sour | 1.2 | rancid |
| 2 | .6 | good | 2.6 | rancid, sour | .8 | rancid |
| 3 | .5 | good | 2.2 | sour | 1.9 | putrid, sour |
| 4 | .5 | good | 3.1 | fair | .9 | putrid |
| 5 | .6 | good | 2.0 | fair, rancid | 1.7 | rancid |
| 6 | .6 | good | 1.6 | rancid, sour | .9 | rancid |
| 7 | .7 | good | 5.0 | rancid | 3.0 | sl. rancid |
| 8 | .9 | good | 2.2 | rancid | 1.2 | sl. rancid |
| 9 | .7 | good | 2.8 | rancid | 1.5 | old |
| 10 | .7 | good | 1.6 | rancid | 1.0 | rancid |

From the data presented it may be readily seen that fatty decomposition occurred in cream even when stored at low temperatures. All samples of normal raw cream contained lipolytic micro-organisms which were capable of causing hydrolysis of fat if conditions favored their growth. These results agree with the findings of Hammer and Collins (5) who showed that lipolytic organisms were common in fresh raw milk and cream. A fat splitting enzyme was also present in all samples studied. Both of these agencies were active throughout a wide range of temperature. There was, however, considerably more hydrolysis of the fat at all temperatures in the samples containing no formaldehyde which indicates that micro-organisms were active in splitting the fat. While considerable variation may be expected in the degree of fat decomposition in cream from different sources, the importance of procuring and processing cream by the creamery soon after it is produced is emphasized.

### EFFECT OF THE GROWTH OF BUTTER CULTURE ORGANISMS AND *L. bulgaricus* ON THE ACID NUMBER OF THE FAT OF CREAM

Most of the butter manufactured in the United States is made from gathered cream, only a comparatively small amount being made from milk separated in creameries. Gathered cream is received by creameries in some areas in a sweet condition while in others it often is excessively sour. In most of the butter producing areas the maximum acidity encountered in

cream is 0.8 to 1.0 per cent and this acid is largely the result of the growth of *S. lactis* organisms. In some sections however cream sometimes develops an acidity considerably in excess of 1.0 per cent. Such an acidity is largely the result of the growth of lactobacilli. Because of poorly organized procurement systems and lax cream grading regulations, cream often remains on farms and in cream stations for prolonged periods before delivery. This situation, coupled with high temperatures, provides conditions suitable for the growth of lactobacilli.

Plant practices often involve the ripening of cream. In this process the acidity may be increased materially before the cream is churned. Under the conditions described the acidity produced is primarily the result of the fermentation of lactose by *S. lactis* which results in the production of lactic acid. The effect of the growth of these homofermentative organisms in cream on the acid number of the fat has been investigated. In trials, portions of sweet cream were sterilized, inoculated with 1 per cent butter culture and ripened at 21° C. to varying acidities. Other lots of cream were inoculated with cultures of *L. bulgaricus* and ripened at 37° C. to acidities considerably above one per cent. The lots of cream were then cooled, churned and the acid numbers of the fat determined.

The data in table 5 shows that even though the acidities of the cream

TABLE 5

*Effect of the growth of butter culture organisms on the acid number of the fat of cream (Cream incubated at 21° C.)*

| Trial I | | Trial II | | Trial III | |
|---|---|---|---|---|---|
| Per cent acidity in cream | Acid number of fat | Per cent acidity in cream | Acid number of fat | Per cent acidity in cream | Acid number of fat |
| 0.11 | 0.60 | 0.09 | 0.55 | 0.11 | 0.65 |
| .39 | .65 | .27 | .50 | .25 | .60 |
| .49 | .60 | .35 | .50 | .35 | .65 |
| .58 | .60 | .60 | .50 | .58 | .65 |
| .80 | .65 | .82 | .55 | .85 | .60 |
| .86 | .60 | .89 | .50 | .88 | .65 |

were increased to the normal maximum of butter culture organisms, which is considerably above the normal churning acidity, the acid number of the fat was not changed appreciably. Likewise, the data in table 6 reveal that the growth of the *L. bulgaricus* failed to alter the acid number of the fat. These results indicate that growth of the common homofermentative organisms in cream is not responsible for increases in the acid number of butterfat and confirm the findings of Orla-Jensen (11) and of Laxa (9) whose data show that ordinary milk souring bacteria had no influence on the acid number of the fat.

TABLE 6

*Effect of the growth of L. bulgaricus on the acid number of the fat of cream (Cream incubated at 37° C.)*

| Trial I | | Trial II | |
|---|---|---|---|
| Per cent acidity in cream | Acid number of fat | Per cent acidity in cream | Acid number of fat |
| 0.10 | 0.70 | 0.13 | 0.45 |
| .88 | .65 | .75 | .50 |
| 1.23 | .65 | 1.30 | .55 |
| 1.60 | .70 | 1.52 | .45 |
| 1.88 | .75 | 2.02 | .45 |

EFFECT OF GROWTH OF CERTAIN LIPOLYTIC ORGANISMS IN CREAM AND BUTTER ON THE ACID NUMBER OF THE FAT

Several organisms which showed definite lipolysis when grown on an agar medium containing fat emulsion were inoculated into portions of sterilized cream. After 7 days incubation at 21° C. the cream samples were churned and the acid numbers of the fat determined.

While all of the organisms showed definite lipolysis on agar plates (table 7), some of them failed to produce rancidity in cream or butter or to cause marked increases in the acid number of the fat. Some of the organisms caused increases in the acid number of the fat and yet failed to produce a typically rancid odor.

Hammer and Collins (5) reported that the highest lipolytic counts were secured on butter that was cheesy rather than rancid. Long (10) also found that certain cultures showing lipolysis on plates containing fat often failed to produce rancidity in butter. Various flavors were produced by the organisms studied, including old, acidy, roquefort, putrid, cheesy and rancid. The acid numbers on the fat of the inoculated samples after incubation ranged from 0.6 to 16.8.

Orla-Jensen (11) showed that certain organisms were able to bring about rancidity and cause high acid values on the fat. In working with pure cultures of *Ps. fragi*, Hussong (6) found that this organism was quite actively lipolytic and caused an increase in the acid number of the fat which was accompanied by a rancid flavor and odor. His work also showed that certain organisms caused marked increases in the acid number of the fat but failed to produce a rancid odor and that many lipolytic organisms are also proteolytic.

It has been shown by many workers that certain micro-organisms have the ability to hydrolyze fat when growing in cream and in butter. A series of trials were made to determine the comparative lipolytic activity of several organisms when growing in cream and in butter. Sweet cream was sterilized in an autoclave, cooled to 21° C., and inoculated with a culture of the organism under consideration. The inoculated cream was well mixed

TABLE 7

*Effect of growth of lipolytic organisms in cream on the acid number of the fat*

| Cream incubated 7 days at 21° C. after inoculation | Per cent acidity in cream | Acid number of fat | Flavor of cream |
|---|---|---|---|
| *P. roqueforti* | 0.32 | 8.6 | roquefort |
| *Myc. lipolytica* | 1.57 | 12.4 | acid, yeasty |
| *Ps. fragi* | .25 | .6 | old |
| *Ps. fluorescens* | .50 | 9.5 | old, putrid |
| *Ach. lipolyticum* | .39 | 9.4 | putrid, rancid |
| *Alc. lipolyticus* | .43 | 16.8 | cheesy, rancid |
| *O. lactis* | .48 | 10.1 | acid |
| Unidentified bacillus A | .30 | 1.8 | putrid |
| Unidentified bacillus B | .53 | 3.5 | putrid |
| Unidentified bacillus C | .32 | 3.4 | putrid, rancid |
| Unidentified bacillus D | .37 | 1.9 | roquefort, rancid |
| Cream before sterilization | .16 | .8 | good |
| Cream after sterilization | .13 | .7 | good, heated |

and divided into two portions. One of these portions was churned, the butter was packed in sterile containers and placed in storage at 5°, 13° and 21° C. The other portion of the inoculated cream was carefully transferred to sterilized fruit jars and placed in storage at the same temperatures as the butter. After 4, 6, 10 and 14 days of storage, samples of cream and butter were removed from storage. The cream was churned and flavor of the butter and acid number of the fat of each sample were determined. In addition the titratable acidity of the cream was determined.

The data showing the lipolytic activity of *Ach. lipolyticum* is presented

TABLE 8

*Comparative lipolytic action of Ach. lipolyticum in cream and in butter*

| Days held at | Cream | | | Butter | |
|---|---|---|---|---|---|
| | Per cent acidity | Flavor | Acid number | Flavor | Acid number |
| *5° C.* | | | | | |
| 0 | 0.13 | good | 1.0 | good | 1.0 |
| 4 | .30 | good | 1.3 | good | 1.3 |
| 6 | .31 | old | 2.0 | old | 1.4 |
| 10 | .32 | old | 2.7 | rancid | 1.6 |
| 14 | .32 | sl. rancid | 3.1 | sl. rancid | 1.7 |
| *13° C.* | | | | | |
| 0 | .13 | good | 1.0 | good | 1.0 |
| 4 | .31 | good | 2.0 | good | 1.6 |
| 6 | .34 | old | 4.3 | off-flavor | 1.8 |
| 10 | .36 | sl. rancid | 4.9 | rancid | 2.4 |
| 14 | .35 | sl. rancid | 5.9 | rancid | 3.1 |
| *21° C.* | | | | | |
| 0 | .13 | good | 1.0 | good | 1.0 |
| 4 | .32 | old | 2.8 | good | 1.9 |
| 6 | .33 | old | 3.3 | sl. rancid | 2.7 |
| 10 | .41 | rancid | 6.2 | rancid | 3.5 |
| 14 | .40 | rancid | 7.1 | rancid | 4.5 |

in table 8. The acid numbers on both the fat of cream and of butter increased progressively throughout the 14 day period. In general the growth at all temperatures was more rapid in cream than in butter as is evidenced by the greater acid numbers on the fat of cream than on the fat of butter under the same holding conditions. The acid numbers in both cream and butter increased most rapidly at the higher temperatures, being at the end of 14 days in the cream 3.1, 5.9 and 7.1 when held at 5°, 13° and 21° C., respectively, and in the butter under the same holding conditions the acid values were 1.7, 3.1 and 4.5. This organism formed comparatively little acid at any temperature, 0.41 per cent being the maximum. In general, off-flavors were evidenced in the cream and butter after about the same period of storage at each temperature regardless of differences in acid numbers.

The results with *Myc. lipolytica* (table 9) were similar to those obtained with *Ach. lipolyticum* with the exception that greater increases in the acid numbers of the fat were observed at all temperatures and all storage periods. After 14 days the acid numbers of the fat of the cream were 34.0, 39.7 and 42.0 at 5°, 13° and 21° C., respectively, and in similarly handled butter the corresponding values were 7.0, 27.9 and 32.6.

*O. lactis* grew more luxuriantly in cream than in butter at all temperatures. This greater growth was evidenced by larger acid numbers on the fat of cream as shown in table 10. After 14 days the acid values in cream were 15.5, 19.4 and 43.0; in butter the corresponding values were 4.1, 14.8 and 38.5. This organism caused greater fatty breakdown in both cream and butter at all temperatures than did the *Ach. lipolyticum* but did not cause as much fat hydrolysis as *Myc. lipolytica* at 5° or 13° C.

The data presented substantiate the statement that the organisms studied, which included common bacterial, mold and yeast species, grew more luxuriantly in cream than they did in butter. The differences were greater at 5° than at 13° and 21° C. It is recognized that the amount of working butter receives influences the rate of growth of the organisms it contains. Since it was impossible to accurately control the degree of working, comparisons between the rate of fat breakdown in the various lots of butter containing different organisms should not be seriously considered. However since all lots of cream were very similar in fat content and other properties it seems logical that comparisons could be made of the lipolytic activity in cream of the various organisms studied.

All of the lots of experimental cream and butter became off-flavored and unmarketable after a few days of storage at all temperatures studied. Using the flavor of the butter and the acid number of the fat as criteria, it may be concluded that all of the organisms studied were extremely damaging to the quality of cream and butter. It may be further concluded that the bacterial species studied, including *Ach. lipolyticum, Ps. fluorescens* and *Alc. lipolyticus* (data on latter two not shown), were less damaging to cream

and butter from the standpoint of fatty decomposition than either *O. lactis* or *Myc. lipolytica.*

TABLE 9

*Comparative lipolytic action of Myc. lipolytica in cream and in butter*

| Days held at | Cream | | | Butter | |
|---|---|---|---|---|---|
| | Per cent acidity | Flavor | Acid number | Flavor | Acid number |
| *5° C.* | | | | | |
| 0 | 0.12 | good | 0.9 | good | 0.9 |
| 4 | .16 | good | 1.0 | good | 1.8 |
| 6 | .17 | good | 3.0 | good | 3.3 |
| 10 | .19 | rancid | 11.7 | sl. rancid | 5.2 |
| 14 | .23 | v. rancid | 34.0 | v. rancid | 7.0 |
| *13° C.* | | | | | |
| 0 | .12 | good | .9 | good | .9 |
| 4 | .21 | old | 7.4 | sl. rancid | 9.9 |
| 6 | .27 | old | 13.8 | rancid | 14.5 |
| 10 | .31 | v. rancid | 18.3 | v. rancid | 21.0 |
| 14 | .35 | v. rancid | 39.7 | v. rancid | 27.9 |
| *21° C.* | | | | | |
| 0 | .12 | good | .9 | good | .9 |
| 4 | .24 | sl. rancid | 7.0 | sl. rancid | 12.0 |
| 6 | .36 | rancid | 13.2 | rancid | 17.3 |
| 10 | .39 | v. rancid | 21.5 | v. rancid | 23.4 |
| 14 | 1.05 | rancid, yeasty | 42.0 | v. rancid | 32.6 |

TABLE 10

*Comparative lipolytic action of O. lactis in cream and in butter*

| Days held at | Cream | | | Butter | |
|---|---|---|---|---|---|
| | Per cent acidity | Flavor | Acid number | Flavor | Acid number |
| *5° C.* | | | | | |
| 0 | 0.12 | good | 0.5 | good | 0.5 |
| 4 | .21 | old | 1.5 | good | .9 |
| 6 | .23 | old | 5.7 | old | 2.3 |
| 10 | .28 | rancid | 13.4 | sl. old | 3.5 |
| 14 | .37 | v. rancid | 15.5 | rancid | 4.1 |
| *13° C.* | | | | | |
| 0 | .12 | good | .5 | good | .5 |
| 4 | .23 | old | 5.3 | good | 1.3 |
| 6 | .29 | old | 8.3 | sl. old | 2.5 |
| 10 | .42 | rancid | 15.3 | sl. rancid | 12.4 |
| 14 | .57 | v. rancid | 19.4 | rancid | 14.8 |
| *21° C.* | | | | | |
| 0 | .12 | good | .5 | good | .5 |
| 4 | .32 | rancid | 8.2 | old | 11.0 |
| 6 | .47 | rancid | 17.5 | old | 14.3 |
| 10 | .68 | v. rancid | 31.3 | v. rancid | 33.4 |
| 14 | 1.05 | v. rancid | 43.0 | v. rancid | 38.5 |

EFFECT OF NEUTRALIZATION OF SOUR CREAM ON THE ACID NUMBER OF THE FAT

Raw cream was inoculated with a culture of a lipolytic organism, *Ach. lipolyticum,* and incubated until the acidity of the cream and the acid number of the fat had increased appreciably. To a series of quart jars, each

containing 1 pound of cream at 30° C., was added neutralizer in increasing amounts so that portions of cream at different acidities were obtained. Sodium carbonate and magnesium oxide were used. The cream was then pasteurized at 62° C. for 30 minutes and cooled. The lots of cream were churned and acid numbers of the fat were determined.

While the data (table 11) reveal a very definite reduction of the free acidity in the fat, the neutralization of this acidity was somewhat slower and less complete than the neutralization of the serum acidity. The free fatty acids were reduced in all samples of cream to which alkali was added but the rate of reduction was slow until the titratable acidity of the cream was reduced appreciably. Using sodium carbonate, comparatively little reduction was noted in the acidity of the fat until the titratable acidity of the cream had been reduced to 0.20 per cent; with magnesium oxide, to 0.15 per cent. After sufficient alkali had been added to reduce the cream to the neutral point using phenolphthalein as an indicator, some free acid still remained in the fat. Although there was no appreciable difference in the degree of reduction of the fatty acids by the two alkalies, magnesium oxide appeared to be slightly more effective than sodium carbonate. Results of experiments by Bird and Breazeale (1) show that a definite reduction in the fatty acids of cream occurred when it was neutralized.

TABLE 11

*Effect of neutralization of sour cream on acid number of the fat*
*(Cream pasteurized at 62° C. for 30 minutes)*

| Treatment of cream | Neutralized with | | | |
|---|---|---|---|---|
| | Sodium carbonate | | Magnesium oxide | |
| | Per cent acidity in cream | Acid number of fat | Per cent acidity in cream | Acid number of fat |
| Raw | 0.72 | 2.1 | 0.72 | 2.1 |
| Pasteurized, not neutralized | .71 | 1.9 | .71 | 1.9 |
| Pasteurized and neutralized | .40 | 1.8 | .44 | 1.8 |
| | .29 | 1.8 | .33 | 1.8 |
| | .20 | 1.4 | .27 | 1.7 |
| | .17 | 1.0 | .15 | 1.2 |
| | .13 | .8 | .11 | 1.2 |
| | .10 | .7 | .10 | 1.2 |
| | .00 | .4 | .00 | .2 |

The work of Clark, *et al.* (2) included a study of the acid numbers of many samples of commercial butter of varying quality. They considered that a good correlation existed between the acid numbers of the fat of butter and the quality of the cream from which it was made and concluded that on the average, the poorer the quality the cream the higher the acid number of the fat of the resulting butter.

In general, the lots of high quality butter had low acid numbers on the

TABLE 12

*Effect of neutralization of sour cream on the acid number of the fat of the resulting cream and butter*
*(All lots of cream from the mixed receipts of a local creamery)*

| | Churning number | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 |
| **Unneutralized raw cream** | | | | | | | | | | |
| Flavor | sour | sour | sour | sour | sour | sour | sour | sour, bitter | sour; sl. rancid | sour, off-flavor |
| Per cent acidity | 0.68 | 0.64 | 0.82 | 0.65 | 0.69 | 0.72 | 0.47 | 0.70 | 0.73 | 0.69 |
| Acid number of fat | 1.5 | 1.9 | 2.5 | 1.5 | 1.3 | 1.2 | 1.4 | 4.1 | 5.9 | 3.7 |
| **Neutralized pasteurized cream** | | | | | | | | | | |
| Flavor | good | good | good | good | good | good | good | sl. bitter | old | old, off |
| Per cent acidity | .25 | .26 | .25 | .27 | .24 | .26 | .25 | .24 | .27 | .23 |
| Acid number of fat | 1.0 | 1.4 | 1.5 | 1.4 | 1.0 | 1.1 | 1.2 | 1.3 | 1.4 | 1.2 |
| **Butter** | | | | | | | | | | |
| Flavor | coarse | coarse | coarse | acidy | old | coarse | clean, coarse | off-flavor | old | coarse |
| Score | 90 | 90.5 | 90 | 89.5 | 90 | 90 | 91 | 89.5 | 89.5 | 89.5 |
| Acid number of fat | .9 | .9 | 1.0 | 1.0 | 1.0 | .9 | 1.0 | 1.7 | 1.4 | 1.0 |

fat. It did not follow however that low quality cream always produced butter with the higher acid numbers on the fat. Poor quality butter made from neutralized cream, often showed a relatively low acid number, depending on the degree of fat hydrolysis, the type and amount of neutralizer used in the cream. Judging from these data and from the results of others (1), one is not justified in assuming that butter with a low acid number on the fat was made from good quality cream.

A study was made on cream delivered to a local creamery, as related to the effect of the neutralization process on the acid number of the butterfat. In each trial the cream was received at the creamery, weighed, sampled and dumped into the pasteurizing vat without regard to quality. When the vat was full, the cream was warmed to about 30° C., a sample of the mixed cream was removed from the vat and immediately cooled. The acidity of the cream in the vat was then standardized to approximately 0.25 per cent with a lime neutralizer. After the cream had been pasteurized and cooled a second sample was removed from the vat. A sample of butter was also taken from the completed churning. The samples of cream were churned and the acid numbers of the fat of the cream as well as of the butter were determined.

The titratable acidities of the unneutralized cream (table 12) varied from 0.47 to 0.82 per cent and the acid numbers of the fat from 1.2 to 5.9. After standardization of the cream with alkali to acidities ranging from 0.23 to 0.27 per cent, the acid numbers of the fat ranged from 1.0 to 1.5. The acid numbers of the fat of the resulting butter ranged from 0.9 to 1.7. In every instance the acid number of the fat was reduced when the acidity of the cream was standardized. While there was considerable variation in the acid numbers of the fat of the unneutralized cream, the acid numbers after neutralization were comparatively uniform. This indicates that the percentage reduction of the fat acidity due to neutralization was considerably greater in the samples of raw cream with high acid numbers than in the samples with low acid numbers. The cream with the high acid numbers before neutralization made slightly lower quality butter than the cream with low acid numbers. Samples 8, 9 and 10 had higher acid numbers than the other samples and the resulting butter was of slightly lower quality.

## CONCLUSIONS

1. Of the two biological agencies causing fat hydrolysis in raw cream, organisms were found to be of greater significance than lipase.

2. In raw cream containing no formaldehyde, in which both lipase and micro-organisms were active, the lipolysis was greater at 5° than at 13° or 21° C.; in cream containing formaldehyde, in which lipase only was active, the degree of hydrolysis increased as the holding temperature of the cream increased within the range studied.

3. The growth of butter culture organisms or *L. bulgaricus* in sterilized

cream, resulting in titratable acidities ranging up to 0.89 per cent with the former and up to 2.02 per cent with the latter, failed to cause changes in the acid numbers of the fat.

4. With the exception of *Ps. fragi*, all organisms studied which showed lipolysis on agar plates containing fat caused increases in the acid numbers of the fat when inoculated into sterilized cream, although rancidity did not result in every instance.

5. All organisms studied were more actively lipolytic in cream than in butter, especially at 5° C.

6. When the titratable acidity of sour cream was reduced by the addition of an alkali, the acid number of the fat was also reduced, but not proportionately.

7. Because of the decrease in the acid number of fat resulting from the neutralization process, it cannot be assumed that butter with a low acid number on the fat was made from good quality cream.

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# LIVE WEIGHT AND MILK-ENERGY YIELD IN HOLSTEIN COWS

W. L. GAINES

*Illinois Agricultural Experiment Station, Urbana, Illinois*

This paper extends an analysis of the relation between live weight and milk-energy yield in cows, based on data available in the literature (*cf.* 1, 2). The records considered here are from the Cornell Station (3, 4) and from the Bureau of Dairy Industry (5). The former pertain to grade Holstein cows of mature age; the latter, to pure-bred half-sister Holstein cows as first-calf heifers and at mature age.

## CORNELL DATA

These represent a feeding trial designed to test the effect of concentrate rations differing in per cent of total protein for 3 successive seasons. They include the initial live weight of each cow and her milk and fat yield for the first 40 weeks of lactation in the first and second seasons and the first 37 weeks in the third season.

*Initial live weight* (*IW*) *and milk energy* (*FCM*). The correlations between initial live weight and milk energy per unit live weight work out as follows:

First season, 32 cows, IW = 1084 to 1333 pounds, $r = -.26 \pm .11$
Second season, 36 cows, IW = 1120 to 1503 pounds, $r = -.06 \pm .11$
Third season, 35 cows, IW = 1062 to 1534 pounds, $r = -.02 \pm .11$

These results suggest a progressive change in the relation of initial live weight to milk-energy yield. Inasmuch as the cows were purchased in the open market shortly before start in the trial it may be inferred that the management in the station herd develops a condition of substantial independence between initial live weight and milk-energy yield per unit initial live weight. That is, milk energy tends to become a multiple of initial live weight under favorable conditions of management.

Twenty-two of the cows were in the trial all 3 seasons. If a progressive change occurs as above suggested it should be indicated more trustworthily by these 22 cows considered by themselves. The correlations for the 22 cows are:

First season, IW = 1084 to 1308 pounds, $r = -.15 \pm .14$
Second season, IW = 1120 to 1503 pounds, $r = +.16 \pm .14$
Third season, IW = 1163 to 1534 pounds, $r = +.11 \pm .14$

Apparently such change as may occur is brought about during the first lactation and rest period in the experiment. As between the cows in this group of 22 during the second and third seasons the larger cows at the start

Received for publication August 3, 1939.

of lactation produce more milk energy per unit size than the smaller cows. If milk energy is expressed as a power function of initial live weight the exponent is greater than unity.

The relation between initial live weight and milk-energy yield for these 22 cows is shown graphically in figure 1.

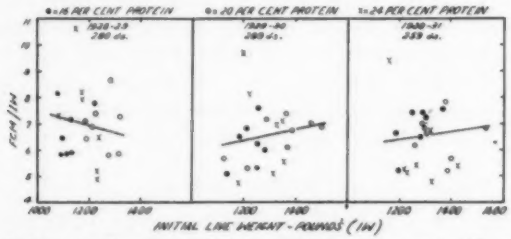


FIG. 1. Relation of milk-energy yield in pounds of 4 per cent milk per pound initial live weight (FCM/IW) to initial live weight in pounds (IW).

Equations of the straight lines:

1928–29, FCM/IW = 10.12 – .00265 IW
1929–30, FCM/IW = 3.99 + .00197 IW
1930–31, FCM/IW = 4.63 + .00141 IW

*Effect of per cent protein in the concentrates.* The general conclusion (3, 4) from the experiment was that there was little difference between the 3 rations as to effect on milk yield. However, on the basis of FCM/IW for the 22 cows in the trials all 3 seasons there appears to be an appreciable and consistent difference in favor of the high-protein concentrates. Table 1 shows the results on this basis. In both the first and second seasons the group average FCM/IW increases with the per cent of protein in the concentrates. In the third season the 16 per cent group, which had received the concentrate with 16 per cent protein continuously in the first and second seasons, was started on 16 per cent concentrates for 5 weeks and then alter-

TABLE 1

*Initial live weight and milk-energy yield per unit initial live weight by groups according to the per cent of protein in the concentrates fed*

(*From publications (3, 4) of the Cornell Station*)

| Group | Number of cows | 1928–29, 280 days | | 1929–30, 280 days | | 1930–31, 259 days | |
|---|---|---|---|---|---|---|---|
| | | Mean IW | Mean FCM/IW | Mean IW | Mean FCM/IW | Mean IW | Mean FCM/IW |
| 16 per cent | 7 | 1125 | 6.74 | 1224 | 6.23 | 1266 | 6.87 |
| 20 per cent | 8 | 1257 | 6.93 | 1326 | 6.54 | 1358 | 6.50 |
| 24 per cent | 7 | 1175 | 7.24 | 1276 | 6.76 | 1269 | 6.00 |

IW = initial live weight in pounds, determined about 3 days after calving.

FCM = milk-energy yield in pounds of 4 per cent milk = .4 × pounds milk + 15 × pounds fat. Milk energy in calories = 340 × FCM.

nated with 24 per cent and then 20 per cent by 5-week periods. A similar procedure was followed for the other two groups. Hence the results are not comparable in the same way as for the first and second seasons.

If milk-energy yield per unit initial live weight is a fair basis of comparison it appears there may be some increase in yield associated with high-protein feeding for the first and second seasons. Results for the third season suggest residual effects. It is the purpose here merely to mention the idea of using FCM/IW as a measure of yield in the first and second seasons.

### BUREAU OF DAIRY INDUSTRY DATA

These records pertain to a breeding project in which a consideration of first importance is a quantitative determination of the milking capacity of the cows for genetic analysis. The Bureau plan is to test each cow with her first calf and again after 5 years of age; feed liberally; milk 3 times daily; breed not earlier than the fifth month of lactation; and use 365 days as the test period. The records are therefore on quite a different plane than those of the Cornell experiment utilized above. The record of live weight is an average of 24 semi-monthly weighings during the test period (in contrast to initial live weight in the Cornell data).

The data considered here are the records of 32 daughters (excluding no.

TABLE 2

*Coefficients of correlation (r) in the records of 32 daughters of a Holstein bull (From a publication (5, table 11) of the Bureau of Dairy Industry)*

| Variables correlated | n | r |
|---|---|---|
| A. Records of the same cow or different cows with respect to: | | |
| 1. Live weight and milk energy per unit live weight | 61 | $-.274 \pm .080$ |
| 2. Live weight and milk energy per unit live weight* | 34 | $+.136 \pm .118$ |
| 3. Live weight and milk energy per unit live weight† | 27 | $-.104 \pm .129$ |
| 4. Live weight and age-corrected milk | 61 | $-.117 \pm .085$ |
| 5. Age and milk energy per unit live weight | 61 | $-.013 \pm .086$ |
| 6. Age and age-corrected milk | 61 | $-.073 \pm .086$ |
| 7. Milk energy and age-corrected milk | 61 | $+.679 \pm .046$ |
| 8. Milk energy and milk energy per unit live weight | 61 | $+.745 \pm .038$ |
| 9. Milk energy per unit live weight and age-corrected milk | 61 | $+.813 \pm .029$ |
| B. First and last records of the same cow with respect to: | | |
| 1. Fat percentage | 22 | $+.624 \pm .088$ |
| 2. Milk energy per unit live weight, FCM/W | 22 | $+.697 \pm .074$ |
| 3. Milk energy, FCM | 22 | $+.725 \pm .068$ |
| 4. Milk | 22 | $+.772 \pm .058$ |
| 5. Age-corrected milk | 22 | $+.826 \pm .046$ |
| C. One record and another of the same cow with respect to: | | |
| 1. Fat percentage | 72 | $+.584 \pm .053$ |
| 2. Milk energy per unit live weight, FCM/W | 72 | $+.692 \pm .042$ |
| 3. Milk energy, FCM | 72 | $+.294 \pm .073$ |
| 4. Milk | 72 | $+.382 \pm .068$ |
| 5. Age-corrected milk | 72 | $+.760 \pm .034$ |

* Records below 1323 pounds live weight, the mean of the 61 records.
† Records above 1323 pounds live weight, the mean of the 61 records.

803) of the Holstein bull, Denton Colantha Sir Rag Apple, at Beltsville as published (5) in table 11.

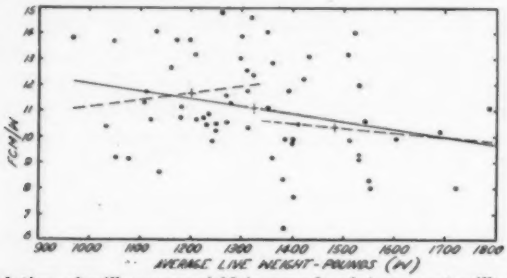


FIG. 2. Relation of milk-energy yield in pounds of 4 per cent milk per pound average live weight (FCM/W) to average live weight in pounds (W). The crosses indicate the means.

Equations of the straight lines:

All records ....., FCM/W = 15.11 − .00304 W
Records above average W, FCM/W = 12.96 − .00177 W
Records below average W, FCM/W = 8.75 + .00244 W

*Average live weight (W) and milk energy (FCM).* Figure 2 presents the correlation surface for W and FCM/W for 61 records of the 32 daughters. The coefficient of correlation is −.274, table 2. Inspection of figure 2 shows an upward trend of FCM/W against W in the left half of the surface. In fact, 34 records below average weight (1323 pounds) give a correlation of +.136; while 27 records above average weight give r = −.104, table 2. Considered over the entire range of live weight FCM/W appears to bear a curvilinear relation to live weight.

The authors (5, p. 7) feel that some of their highest-producing cows could have made still greater records with 4 daily milkings, artificial aids to cooling of the hard-working, heat-producing organism during the hot days of summer, etc. Since the highest-producing cows tend to be found among the largest cows we may infer that the full capacity of the largest cows was not realized, and had it been we might find substantially a horizontal trend of FCM/W against W for the whole range of W. Without artificial cooling there are times when an 1800-pound cow, as compared with a 900-pound cow, is decidedly handicapped by the work of getting rid of surplus heat.

As the record stands the daughters of this bull seem to have inherited a very strong proclivity to lactation, manifest not alone in the absolute milk energy produced but also in the relation of milk-energy yield to live weight. Figure 3 affords a direct graphic demonstration of how well the individual observations conform to a simple-multiple relation between live weight and milk-energy yield.

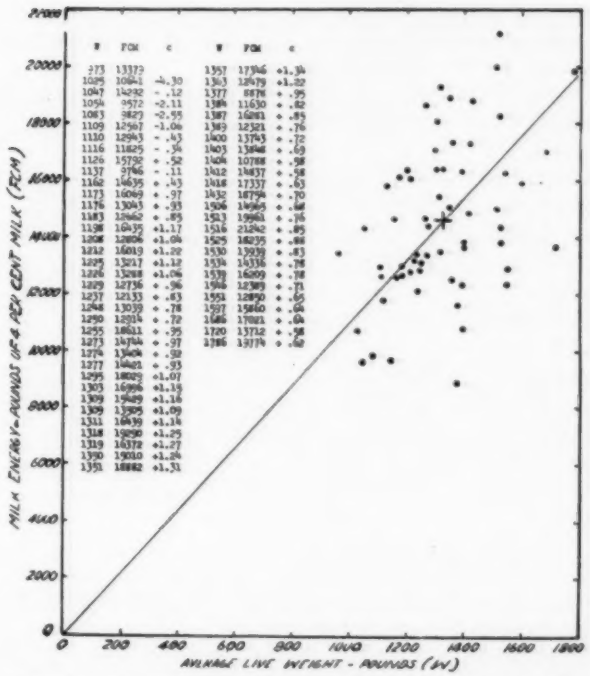


FIG. 3. Relation of milk-energy yield in pounds of 4 per cent milk (FCM) to average live weight in pounds (W).

The straight line is drawn from the origin through the means (cross) and has the equation, FCM = 11.02 W (mean FCM = 14581, mean W = 1323, and 14581/1323 = 11.02).

The numerical data on the chart give the observations arranged in order of live weight and fitted with the equation, $FCM = bW^c$, by live-weight stages. For the lowest two live weights $c = -4.30$; for the lowest 3, $c = -.12$; and so on to all 61 observations where $c = +.62$. For live weights from 973 to 1116 pounds, inclusive, milk-energy yield is inversely proportional to the .34 power of live weight. This may be interpreted to mean that there is a tendency for the cows with high rate of yield to have low rate of gain in live weight, thus tending to associate high yield with low-average live weight. The use of initial live weight, instead of average live weight, might eliminate this difficulty. (The published data do not include initial live weight.) For live weights from 973 to 1357 pounds, inclusive, milk-energy yield is proportional to the 1.34 power of live weight. Beyond this point there are rapid declines in the value of c, due to several exceptionally low yields per unit live weight. This may be interpreted to indicate a relative environmental handicap for the large cows. These interpretations consider that age of the cow is of itself a practically negligible factor in her milk-energy yield.

*Age correction vs. FCM/W.* The age corrections applied to milk yield (5, p. 25) "are a composite of the available adjustment factors." (The number of sets of factors in the composite is not stated but it must be large

to include all of those available.) As shown in table 2 the correlation between age and age-corrected milk is – .07. That is, the correction effectively reduces the age trend to a horizontal line.

The correlation between age and FCM/W is – .01. That is, FCM/W, a direct factual measure of yield, is even more independent of age than is age-corrected milk.

The question may be raised, is age correction an indirect way of allowing for live weight, or is FCM/W an indirect way of allowing for age? A definite answer is afforded by the data of Kleinberg previously cited (2), representing some 14,000 annual records. When live weight was held within a narrow range age had no influence on milk yield. On the other hand when age was held within a narrow range, live weight had practically the same effect on milk yield as when age was not held constant. It may be concluded that age correction is an indirect way of allowing for live weight.

While age-corrected milk and FCM/W are closely related, r = .813, the two things are by no means identical or the same in meaning. The close association arises in the condition that both are largely dependent on the absolute milk-energy yield, as evidenced by the correlations given in table 2, A7 and A8.

*Correlation between records of the same cow.* Dealing with the first and last records of the same cow, with respect to the same item, table 2, section B, the items and correlations, in increasing order of r follow: fat percentage, .62; FCM/W, .70; FCM, .73; milk, .77; age-corrected milk, .83. What is the meaning of high and low values of r in such a comparison?

Let us consider fat percentage to illustrate one point of view in answer to the question. If the 22 cows are inherently exactly alike fat percentage will fluctuate at random, from non-inherent influences, and the correlation will tend to be zero; if the cows are inherently or consistently unlike the correlation will tend to be one. Conversely, a low value of r indicates likeness (presumably genetic homogenity) in the cows, while a high value of r indicates unlikeness in the cows, with respect to the character under consideration. As an illustration, the 22 grade Holstein cows, at Cornell, table 1, purchased on the market without regard to relationship, show a correlation for the first and last records with respect to fat percentage of r = .93, in contrast to r = .62 for the comparable correlation in the 22 pure-bred Holstein cows at Beltsville, table 2, all daughters of the same sire. This example, at least, seems consistent with the above interpretation of the meaning of high and low values of r in the correlation between the first and last records of the same cow with respect to fat percentage.

Age correction changes the correlation for milk from .77 to .83, that is, makes the cows more unlike. Dividing by W changes the correlation for FCM from .73 to .70, that is, makes the cows more alike. Unfortunately,

there seems no way to know which direction of change better fits the true condition of likeness.

## SUMMARY

Sixty-six records of 22 grade Holstein cows in a feeding experiment at Cornell for 3 successive seasons, show that milk-energy yield per unit initial live weight decreased with live weight during the first season, and increased with live weight during the second and third seasons. It is concluded that milk-energy yield tends to be a multiple of initial live weight (3 days after calving) under continued favorable, practical conditions.

Sixty-one records of 32 daughters of the same Holstein bull in a breeding experiment at Beltsville show that milk-energy yield per unit average live weight decreased with live weight; 27 of the 61 records above average live weight show a similar tendency; 34 of the 61 records below average live weight show an opposite tendency. The 61 records show milk-energy yield per unit live weight is independent of age ($r = -.01 \pm .09$); age-corrected milk is independent of age ($r = -.07 \pm .09$). It is concluded that age-correction is an indirect way of allowing for live weight.

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(3) HARRISON, E. S., AND SAVAGE, E. S. The effect of different planes of protein intake upon milk production. Cornell Univ. Agr. Exp. Sta. Bull. 540. 1932.

(4) HARRISON, E. S., SAVAGE, E. S., AND WORK, S. H. The effect of different planes of protein intake upon milk production. II. Cornell Univ. Agr. Exp. Sta. Bull. 578. 1933.

(5) FOHRMAN, M. H., AND GRAVES, R. R. Experiments in breeding Holstein-Friesian cattle for milk- and butterfat-producing ability, and an analysis of the foundation cows and of the first outbred generation. U. S. Dept. Agr. Tech. Bull. 677. 1939.

to include all of those available.) As shown in table 2 the correlation between age and age-corrected milk is – .07. That is, the correction effectively reduces the age trend to a horizontal line.

The correlation between age and FCM/W is – .01. That is, FCM/W, a direct factual measure of yield, is even more independent of age than is age-corrected milk.

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# THE VIABILITY OF SEEDS AS AFFECTED BY THE SILOING PROCESS

T. E. WOODWARD

*U. S. Department of Agriculture, Bureau of Dairy Industry*

Seeds of some of the farm crops and of the more common weeds were placed in the silo along with corn or alfalfa at the time of filling. When the silage was fed down to the seeds they were removed, dried at room temperature, and tested for germination by the Division of Seed Investigations, Bureau of Plant Industry. Twenty-nine lots (or kinds) of seed were buried in silage for the study. Duplicate samples of 22 lots were stored in the office and tested for germination to compare their viability with that of the samples in the silo. Detailed results of this work are shown in the following tables.

TABLE 1

*Viability of seeds buried in alfalfa silage and in grass-and-alfalfa silage, and of similar seeds stored in the office*

| Name of seed | Place stored | Duration of test | Germination | Condition of remaining seeds at the close of the test |
|---|---|---|---|---|
| (Seeds in alfalfa silage with 34 per cent moisture, 14 feet below the surface, from June 1, 1934, to January 18, 1935) | | | | |
| | | *Days* | *Per cent* | |
| Shepherd's purse[1] | Silo | 43 | 0 | Many sound |
| | Office | 43 | 27 | Many sound |
| Chickweed[2] | Silo | 24 | 0 | |
| | Office | 24 | 97 | |
| (Seeds in grass-and-alfalfa silage with 45 to 50 per cent moisture from May 28, 1936, to July 25, 1936) | | | | |
| | | *Days* | *Per cent* | |
| Chickweed | Silo | 28 | 0 | |
| | Office | 63 | 88 | |
| Buttercup[3] | Silo | 49 | 0 | |
| | Office | 49 | 87 | |
| Dandelion[4] | Silo | 21 | 0 | |
| | Office | 21 | 52 | |
| Shepherd's purse[1] | Silo | 28 | 0 | |
| Timothy[5] | Silo | 12 | 0 | |
| | Office | 12 | 96 | |
| Oats[6] | Silo | 8 | 0 | |
| | Office | 8 | 77 | |

[1] *Bursa bursa-pastoris.*
[2] *Alsine media.*
[3] *Ranunculus* sp.
[4] *Taraxacum officinale.*
[5] *Phleum pratense.*
[6] *Avena sativa.*

When the 29 lots of seed were removed from the silo and tested, all seeds failed to germinate except some of those in the samples of *Lespedeza sericea,*

Received for publication September 5, 1939.

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| Name of seed | Place stored | Duration of test | Germination | Condition of remaining seeds at the close of the test |
|---|---|---|---|---|
| (Seeds in alfalfa silage with 34 per cent moisture, 14 feet below the surface, from June 1, 1934, to January 18, 1935) | | | | |
| | | *Days* | *Per cent* | |
| Shepherd's purse[1] | Silo | 43 | 0 | Many sound |
| | Office | 43 | 27 | Many sound |
| Chickweed[2] | Silo | 24 | 0 | |
| | Office | 24 | 97 | |
| (Seeds in grass-and-alfalfa silage with 45 to 50 per cent moisture from May 28, 1936, to July 25, 1936) | | | | |
| | | *Days* | *Per cent* | |
| Chickweed | Silo | 28 | 0 | |
| | Office | 63 | 88 | |
| Buttercup[3] | Silo | 49 | 0 | |
| | Office | 49 | 87 | |
| Dandelion[4] | Silo | 21 | 0 | |
| | Office | 21 | 52 | |
| Shepherd's purse[1] | Silo | 28 | 0 | |
| Timothy[5] | Silo | 12 | 0 | |
| | Office | 12 | 96 | |
| Oats[6] | Silo | 8 | 0 | |
| | Office | 8 | 77 | |

[1] *Bursa bursa-pastoris.*
[2] *Alsine media.*
[3] *Ranunculus* sp.
[4] *Taraxacum officinale.*
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When the 29 lots of seed were removed from the silo and tested, all seeds failed to germinate except some of those in the samples of *Lespedeza sericea,*

Received for publication September 5, 1939.

TABLE 2

*Viability of seeds buried in corn silage, and of similar seeds stored in the office*
(Seeds in corn silage with normal moisture content, 30 feet below surface, from October 5, 1936, to April 4, 1937)

| Name of seed | Place stored | Duration of test | Germination | Condition of remaining seeds at the close of the test |
|---|---|---|---|---|
| | | *Days* | *Per cent* | |
| Bigseed ladys-thumb or Pennsylvania smartweed[1] | Silo | 148 | 0 | 4 per cent sound |
| | Office | 148 | 37 | 41 per cent sound |
| Goose grass[2] | Silo | 21 | 0 | |
| | Office | 21 | 97 | |
| Amaranth[3] | Silo | 148 | 0 | |
| | Office | 127 | 68 | 21 per cent sound |
| Crabgrass[4] | Silo | 56 | 0 | |
| | Office | 56 | 85 | Decayed |
| Common ragweed[5] | Silo | 117 | 0 | |
| | Office | 148 | 74 | None sound |
| Pigeon grass or foxtail[6] | Silo | 149 | 0 | |
| | Office | 148 | 93 | |

[1] *Polygonum pennsylvanicum.*
[2] *Eleusine indica.*
[3] *Amaranthus sp.*
[4] *Digitaria sp.*
[5] *Ambrosia artemisiifolia.*
[6] *Setaria sp.*

bindweed, and American dragonhead mint. There was a high percentage of hard seeds in the samples of *Lespedeza sericea* and bindweed and a very small percentage in the samples of sweetclover seed that survived the ensiling process and the germination test.

Some seeds germinated in all the 22 lots that were kept in the office, except in those of perennial sowthistle and Canada thistle, and even these two lots were known to be viable shortly before they were placed in the silo. Although no definite statement can be made regarding the viability of the 7 lots for which no duplicates were kept in the office, it should be noted in table 1 that one sample of shepherd's purse seed kept in the office germinated to the extent of 27 per cent, which indicates that the other sample of shepherd's purse might likewise have contained some viable seed. Also, in the 1938 trial no chickweed was kept in the office (table 3), but in previous trials two samples kept in the office had a high percentage of germination (table 1). Our general knowledge of three others—*Lespedeza sericea,* Johnson grass, and sweetclover—would lead us to believe that these would show some viability after being kept in the office. Some of the two remaining lots of seeds—Indian mustard and American dragonhead mint—were successfully used to raise plants in a greenhouse shortly before seeds were placed in the silo. It appears reasonable to assume, therefore, that they would have shown some viability if they had been tested after storage in an office.

Silages of corn, and grass and alfalfa all destroyed the viability of the seeds and while there is no way of estimating the relative effectiveness of the different silages in this respect, there is a slight indication that the low-

TABLE 3

*Seeds[1] in alfalfa silage with different moisture contents and preserved with and without molasses compared with seeds kept in the office. Seeds placed in the silo June 1938*

| Name of seed | Duration of test | 32 per cent moisture no molasses 6 feet below surface removed December 1938 | | 41 per cent moisture 4.18 per cent molasses 14 feet below surface removed January 1939 | | 48.5 per cent moisture no molasses 15½ feet below surface removed January 1939 | |
|---|---|---|---|---|---|---|---|
| | | Germination | Condition of remaining seeds at close of test | Germination | Condition of remaining seeds at close of test | Germination | Condition of remaining seeds at close of test |
| | *Days* | *Per cent* | | *Per cent* | | *Per cent* | |
| Quackgrass[2] | 21 | 0 | Decayed | 0 | Decayed | 0 | Decayed |
| Corn cockle[3] | 14 | 0 | " | 0 | " | 0 | " |
| India mustard[4] | 7 | 0 | " | 0 | " | 0 | " |
| Perennial sowthistle[5] | 14 | 0 | " | 0 | " | 0 | " |
| Oxeye daisy[6] | 14 | 0 | " | 0 | " | 0 | " |
| Chickweed[7] | 21 | 0 | " | 0 | " | 0 | " |
| Johnson grass[8] | 28 | 0 | " | 0 | " | 0 | " |
| Lespedeza sericea[9] | 21 | 1 | 56% hard seed; rest decayed | 5 | 47% hard seed; rest decayed | 2 | 60% hard seed; rest decayed |
| Bindweed[10] | 28 | 11 | 40% hard seed; rest decayed | 9 | 37% hard seed; rest decayed | 5 | 44% hard seed; rest decayed |
| Sweet clover[11] | 7 | 0 | No hard seed; decayed | 0 | 1% hard seed; rest decayed | 0 | 1% hard; rest decayed |
| American dragonhead mint[12] | 28 | 3 | | 36 | | 0 | Decayed |
| Canada thistle[13] | 14 | 0 | Decayed | 0 | Decayed | 0 | " |
| Horse nettle[14] | 14 | 0 | " | 0 | " | 0 | " |
| Leafy spurge[15] | 14 | 0 | " | 0 | " | 0 | " |
| Perennial pepper grass or white-top[16] | 14 | 0 | " | 0 | " | 0 | " |

[1] Most of these seeds were furnished by Mr. L. W. Kephart, Bureau of Plant Industry.
[2] *Agropyron repens.*
[3] *Agrostemma githago.*
[4] *Brassica juncea.*
[5] *Sonchus arvensis.*
[6] *Chrysanthemum leucanthemum pinnatifidum.*
[7] *Alsine media.*
[8] *Sorghum halepense.*

TABLE 3.—(*Continued*)

| Name of seed | Duration of test | 69 per cent moisture no molasses 17 feet below surface removed February 1939 | | 65 per cent moisture 3.44 per cent molasses 21 feet below surface removed March 1939 | | Kept in office | | |
|---|---|---|---|---|---|---|---|---|
| | | Germination | Condition of remaining seeds at close of test | Germination | Condition of remaining seeds at close of test | Duration of test | Germination | Condition of remaining seeds at close of test |
| | *Days* | *Per cent* | | *Per cent* | | *Days* | *Per cent* | |
| Quackgrass[2] | 21 | 0 | Decayed | 0 | Decayed | 30 | 22 | Decayed |
| Corn cockle[3] | 14 | 0 | " | 0 | " | 21 | 87 | |
| India mustard[4] | 7 | 0 | " | 0 | " | No test made | | |
| Perennial sowthistle[5] | 14 | 0 | " | 0 | " | 21 | 0 | Decayed |
| Oxeye daisy[6] | 14 | 0 | " | 0 | " | 30 | 51 & 30 | " |
| Chickweed[7] | 21 | 0 | " | 0 | " | No test made | | |
| Johnson grass[8] | 28 | 0 | " | 0 | " | No test made | | |
| Lespedeza sericea[9] | 21 | 1 | 62% hard seed; rest decayed | 0 | 67% hard seed; rest decayed | No test made | | |
| Bindweed[10] | 28 | 6 | 49% hard seed; rest decayed | 5 | 53% hard seed; rest decayed | 21 | 22 | 53% hard seed; rest decayed |
| Sweet clover[11] | 7 | 0 | 1% hard seed; rest decayed | 0 | 1% hard seed; rest decayed | No test made | | |
| American dragonhead mint[12] | 28 | 0 | Decayed | 0 | Decayed | No test made | | |
| Canada thistle[13] | 14 | | " | 0 | " | 21 | 0 | Decayed |
| Horse nettle[14] | 14 | 0 | " | 0 | " | 30 | 55 | " |
| Leafy spurge[15] | 14 | 0 | " | 0 | " | 30 | 59 | " |
| Perennial pepper grass or white-top[16] | 14 | 0 | " | 0 | " | 30 | 33 | " |

[9] *Lespedeza sericea.*
[10] *Convolvulus arvensis.*
[11] *Melilotus officinalis.*
[12] *Dracocephalum parviflorum.*
[13] *Cirsium arvense.*
[14] *Solanum carolinense.*
[15] *Euphorbia Esula.*
[16] *Lepidium draba.*

moisture silages may not be quite so effective in destroying the viability as high-moisture silages.

One should not overlook the fact that at the conclusion of the germination test some of the seeds were rated as "sound" or "hard." Such seeds may or may not have germinated subsequently, if they had been subjected to conditions different from those under which these tests were made.

It is well known that when a weedy crop is made into hay, many weed seeds are preserved in a viable condition, also that the application on the land of stable or barnyard manure, at least that which has not been composted or allowed to rot, usually increases the prevalence and growth of weeds. It appears, therefore, that the conversion of weedy crops into silage instead of into hay will help materially in the control of weeds.

## American Dairy Science Association Announcements

Thirty-Fifth Annual Meeting, Purdue University, West Lafayette, Indiana, June 24–28, 1940. Information concerning rooms and housing will be given in the April issue of this Journal.

### CALL FOR TITLES AND ABSTRACTS

All abstracts to be presented at our annual meeting in June must be in our hands by April 15. Please send titles and abstracts to Dr. B. E. Horrall, Department of Dairy Husbandry, Purdue University, West Lafayette, Indiana.

VOLUME XXIII MARCH, 1940 NUMBER 3

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Zeitschrift für Untersuchtung der Lebensmittel
Zeitschrift für Züchtung. Reihe B. Tierzuchtung und Zuchtungbiologie
Zentralblatt für Bacteriologie
Züchtungskunde

## SPECIAL PUBLICATIONS

Federal Dairying and Bacteriological Establishment, Liebefeld, Berne, Switzerland

International Association of Ice Cream Manufacturers
International Association of Milk Dealers

National Institute for Research in Dairying, Reading, England
New York Association of Dairy and Milk Inspectors

Prussian Dairy Research Institute, Kiel, Germany

State Agricultural Colleges and Experiment Stations

The Royal Technical College, Copenhagen, Denmark

United States Department of Agriculture

# ABSTRACTS OF LITERATURE

## BACTERIOLOGY

**96. The Decomposition of Citric Acid by Betacoccus cremoris.** G. VAN BEYNUM AND G. W. PETTE. State Agr. Exp. Sta., Hoorn, Netherlands. J. Dairy Res., *10:* 250–266, 1939.

Analyses reveal that the fermentation products of *Betacoccus cremoris* in neutral milk are acetic acid and carbonic acid. In acidified milk or in mixed cultures of this bacterium and lactic acid streptococci the products are acetic acid, $CO_2$, diacetyl, acetylmethyl carbinol and 2–3 butylene glycol. Diacetyl is only formed when an oxidation with atmospheric oxygen can take place. Carbinol is found in aerobic and in anaerobic cultures. It may be reduced to butylene glycol. All of these substances are formed from the citric acid of the milk. From 1 mol. of citric acid are formed: 2 mol. of $CO_2$, 1–1.5 mol. of acetic acid, 0.5–0 mol. of $C_4$ compounds. A reciprocal relationship is shown to exist between the quantities of acetic acid and $C_4$ compounds. When the amount of acetic acid is high the $C_4$ compounds content is low. The higher the acidity of the medium in which the betacocci are cultivated the higher is the amount of $C_4$ compounds. The authors assume that pyruvic acid is an intermediate product and present formulae showing the decomposition of citric acid. S.T.C.

**97. The Nutritional Requirements of the Lactic Acid Bacteria.** J. G. DAVIS, Nat. Inst. for Res. in Dairying, Univ. of Reading, Reading, England. J. Dairy Res., *10:* 186–201, 1939.

Serum and blood were found not to have a marked effect on the growth of lactic acid bacteria in milk. Heated serum and, to a greater extent, heated blood usually stimulated growth. Unheated serum was found to inhibit *S. lactis,* when a small inoculum of washed cells was used. Under practical conditions no effect was noticed. The author concludes, therefore, that "it is unlikely that 'slow starter', is ever attributable to infiltration of serum or the constituents of serum." S.T.C.

## BREEDING

**98. Biennial Reviews of the Progress of Dairy Science.** Section G, Genetics of Dairy Cattle. J. Dairy Res., *10:* 370–393, 1939.

A review of recent work relative to the genetics of dairy cattle.

S.T.C.

## BUTTER

**99. Bacteriological Testing of Butter. New and Simplified Routine Methods.** G. M. MOIR AND R. R. RUSSELL. New Zealand Dept. of Agr., Wallaceville, Wellington. J. Dairy Res., *10:* 310–325, 1939.

Microplate cultures are prepared to obtain counts of total bacteria, "heat resistance" bacteria, lypolytic organisms, yeasts and molds in butter. The method for counting colonies of lypolytic bacteria is based upon their ability to produce around each colony a halo of white solid fatty acids in place of the clear glistening fat globules. S.T.C.

**100. Oxidation of the Fat of Butter During Cold Storage.** W. J. WILEY, Dairy Res. Inst., Palmerston North, New Zealand. J. Dairy Res., *10:* 300–309, 1939.

The oxidation of the fat of butter during cold storage was studied by measuring the fat-aldehyde value of the fat. Acidity, starter organisms, salt and low-pasteurization temperatures were found to favor oxidation. Neither diacetyl nor acetoin influenced the oxidation.

The author postulates the presence in ripened pasteurized cream and in unripened raw cream of a fat-oxidizing enzyme which is most active at low pH values, and high salt concentrations. S.T.C.

**101. Observation on Epidemics of Moldy Salted Butter.** V. L. TURGASEN, Armour & Co., Chicago. Nat. Butter and Cheese J., *30:* 11, 44, 1939.

Seven epidemics are described and their causes are indicated. Sources of contamination in order of importance are unclean equipment, air-borne infection, insufficient pasteurization exposures of the cream, contaminated tubs, boxes and liners and contamination from such sources as water, salt and butter cultures. W.V.P.

## CHEESE

**102. The Relation of Certain Lactic Acid Bacteria to Open Texture in Cheddar Cheese.** I. R. SHERWOOD, Dairy Res. Inst., Palmerston North, New Zealand. J. Dairy Res., *10:* 326–335, 1939.

The rate of evolution of carbon dioxide from cheese was measured by determining the ml. of N/10 barium hydroxide neutralized by the gas from 25 g. of cheese in two days at room temperature. "Open" cheese was shown to evolve carbon dioxide much more rapidly than "close" cheese. From open cheese lactobacilli or betacocci capable of producing carbon dioxide relatively rapidly were isolated. The addition of such organisms to cheese milk resulted in the development of slit openness in the cheese. S.T.C.

**103. A Simple and Accurate Viscosimetric Form of Rennet Test.** C. W. KING AND E. M. MELVILLE, The West of Scotland Agr. College, Glasgow. J. Dairy Res., *10:* 340–354, 1939.

A viscosity type of rennet test is described. A mathematical treatment of the problems involved in the use of the method is given. S.T.C.

**104. The Assessment of Curd Firmness Prior to Cutting.** F. M. V. COPPEN, Nat. Inst. for Res. in Dairying, Univ. of Reading, Reading, England. J. Dairy Res., *10:* 336–339, 1939.

Using the method devised by Scott Blair (J. Dairy Res., *9:* 347. 1938) the "firmness" of cheese curds immediately prior to cutting was determined and related to an expert's opinion. The smallest difference in modulus which could be correctly appreciated by the cheese maker was of the order of 8 per cent. Normal curds, regardless of type, were softer in the summer months than in the winter. S.T.C.

**105. The Making of Processed Cheese.** H. H. SOMMER AND H. L. TEMPLETON, Univ. of Wis., Madison. Wisconsin Agr. Exp. Sta. Res. Bull. 137, June, 1939.

Processing by selecting raw cheese, blending, grinding, heating and packaging gives uniformity of quality and a desirable package. Some patents restricting manufacture of process cheese have expired. Historical background of the industry and legal definitions are given. The processing procedure is described including details of selecting and blending of cheese to obtain desired flavor, body properties and composition; trimming and grinding to remove inedible portions and to prepare the cheese for effective heating; heating and stirring to obtain a homogenous and fluid mixture for packaging; use of emulsifiers; and packaging. Processing and packaging machines are described. Preparation of cheese spreads is given. Factors to consider in planning cheese processing operations are presented to indicate that approximately 100,000 lbs. of raw cheese is necessary to permit proper blending operations and successful control of quality when the output is 1,000 pounds per day and the average age of cheese is 3 to 4 months; that the minimum investment in equipment would be about $1500 to $2000; and that efficient large scale operations require considerable additional investment in labor-saving machinery. One table, 5 figures, 20 references and a list of 102 U. S. patents showing date of issue, patentee and patent title.

W.V.P.

## CHEMISTRY

**106. The Rapid Determination of Peroxide Values for the Fat in Milk Powders.** J. A. B. SMITH, The Hannah Dairy Res. Inst., Kirkhill, Ayr. J. Dairy Res., *10:* 294–299, 1939.

A method which although yielding only about 90 per cent of the true value is considered of value by the author due to its rapidity is described.

Ten g. of milk powder are weighed into a 100 ml. graduated flask and 50 ml. glacial acetic acid added. The mixture is warmed to 35° C. for 5 minutes and shaken at intervals during that time. Chloroform is added from a burette until the 100 ml. mark is reached, and during this addition the mixture is well-shaken several times. The mixture is filtered rapidly through an ordinary filter funnel. An aliquot of the filtrate is measured out, one ml. of a saturated solution of potassium iodide added and the mixture shaken for one minute. It is then diluted with 100 ml. of water and titrated with standard sodium thiosulphate, 0.01 to 0.002 normal.

S.T.C.

**107. The Preparation of Phenyl Phosphoric Esters.** E. J. KING AND T. F. NICHOLSON, British Postgraduate Medical School, London. Biochem. J., *33:* 1182, 1939.

In this paper are presented methods for conveniently preparing several phenyl phosphoric esters. The use of these in the phosphatase test of milk is not discussed but they may be conveniently adopted. The procedures described are for the preparation of barium phenyl phosphate, disodium phenyl phosphate, ortho-methylphenyl phosphate, para-bromphenyl phosphate, para-nitrophenyl phosphate, cyclo hexanol phosphate.

K.G.W.

**108. The Rates of Enzymic Hydrolysis of Phosphoric Esters.** E. J. KING AND G. E. DELORY, British Postgraduate Medical School, London. Biochem. J., *33:* 1185, 1939.

The relative rates of hydrolysis of several phosphoric esters is presented. The rate of hydrolysis and the optimum pH for hydrolysis appear to increase with increasing acidity of the phosphoric ester. K.G.W.

**109. Estimation of Lactic Acid in Biological Material by Oxidation with Ceric Sulfate.** J. J. GORDON AND J. H. QUASTEL, Biochemical Laboratory, Cardiff City Mental Hospital, Cardiff, Wales. Biochem. J., *33:* 1332, 1939.

A method is presented for estimating lactic acid in biological materials using ceric sulfate. The acetaldehyde resulting from the oxidation of lactic acid is absorbed by sodium bisulphate and estimated iodometrically. The error in estimation of lactic acid does not exceed 5 per cent. K.G.W.

**110. Determining Riboflavin. A Fluorometric and Biological Method.** G. C. SUPPLEE, R. C. BENDER, AND O. G. JENSEN, The Borden Co., Bainbridge, N. Y. Ind. Eng. Chem., Analyt. Ed., *11:* 9, 495–498, 1939.

Methods for extracting and preparing riboflavin solutions from various concentrates, adsorbates, whey and miscellaneous products and for the fluorometric examination of the riboflavin solutions in "black light" are presented. A method for the biological determination of riboflavin is given and the results obtained by both methods are compared. The methods show a correlation greater than 90 per cent when applied to dry yeast and raw peanuts, 80 to 85 per cent when applied to alfalfa meal and liver and less than 50 per cent for soy bean and corn meals. The methods may be used interchangeably for certain types of riboflavin solutions and adsorbates. The fluorometric method gives check results within 10 per cent variation from independent determinations with different operators and within 3 to 5 per cent with experienced operators. B.H.W.

111. **Determining Riboflavin in Dried Milk Products.** ROYAL A. SULLIVAN AND L. C. NORRIS, Cornell Univ., Ithaca, N. Y. Ind. Eng. Chem., Analyt. Ed., *11:* 10, 535–540, 1939.

Riboflavin is extracted from dried milk products by refluxing with a dilute solution of acid in 75 per cent acetone. Unstable colored impurities are removed by the addition of hydrogen peroxide to the solvent. The resulting solution is neutralized, filtered and used for the determination of riboflavin with a photoelectric photometer. Ninety per cent of the color of riboflavin is removed by reduction with sodium hyposulfite. By observation of the light absorption before and after reduction the concentration of the solution is calculated to $\pm$ 0.05 microgram per l. The photometer is calibrated with pure synthetic riboflavin. The photometer wiring diagram, a transmission curve for the light filters used, and other pertinent data are presented. B.H.W.

## CONCENTRATED AND DRY MILK; BY-PRODUCTS

112. **Bacteriological Studies of Canned Milk Products.** AGNES A. NICHOLS, The Hannah Dairy Res. Inst., Kirkhill, Ayr. J. Dairy Res., *10:* 231–249, 1939.

A large number of evaporated milk and canned cream samples from general sources and from 3 factories in Scotland were examined. One hundred per cent of the tins examined from general sources were sterile. The factory samples, especially those of canned cream, had a lower sterility percentage, although it was considered that this was due to special circumstances of operation, and that ordinarily a sterile pack should be obtained. An outbreak of "buttons" in sweetened condensed milk was attributed to three causative organisms, one a species of *Actinomyces,* the other two species of the genus *Penicillum.* S.T.C.

113. **Bacteriological Studies of Spray-Dried Milk Powder.** AGNES A. NICHOLS, The Hannah Dairy Res. Inst., Kirkhill, Ayr. J. Dairy Res., *10:* 202–230, 1939.

Data are presented on over 400 samples of spray-dried milk powder secured from eight factories operating in England and Scotland. Plate counts varied from 1400 to 149,000,000 per gram. About 10 per cent of the samples tested in one ml. quantities gave positive presumptive coliform tests. The desirability of establishing grades for spray-dried milk powder is discussed. S.T.C.

## DISEASE

114. **Biennial Reviews of the Progress of Dairy Science.** Section F, Milk Borne Diseases. J. Dairy Res., *10:* 355–369, 1939.

A review of recent work relative to milk borne diseases. S.T.C.

115. **The Casein Number for Diagnosis of Mastitis. II. The Effect of Advanced Lactation and of Storage and Preservation of Milk Sample.** S. G. ROWLAND AND M. ZEIN-EL-DINE, Univ. of Reading, Reading, England. J. Dairy Res., *10:* 267–271, 1939.

The casein number (percentage of casein nitrogen in total nitrogen of the milk) method of mastitis diagnosis was found not applicable to cows in advanced lactation.

Storage of milk samples for more than a few hours at 16–18° C. resulted in a decrease in the casein number. Storage at 0–2° C., and at 16–18° C. with the addition of 0.2, 0.05, and 0.01 g. potassium dichromate, formalin or mercuric chloride respectively per 100 ml. of milk, prevented significant reduction of the casein number for 24–48 hours. S.T.C.

## FEEDS AND FEEDING

116. **The Nutritive Value of Proteins for Milk Production. V. The Effect of High Temperature and of Season on the Nutritive Value of Grass Proteins, the Supplementary Effect of the Maintenance Ration on the Production Ration, and the Effect of Feeding a High-Protein Ration.** S. MORRIS AND S. C. RAY, The Hannah Dairy Res. Inst., Kirkhill, Ayr. J. Dairy Res., *10:* 165–185, 1939.

As measured by milk production the nutritive value of artificially dried spring grass was unaffected by the temperature of drying even to the extent of scorching the grass. Dried spring grass was found to have a higher and later autumn grass a lower biological value than early autumn grass. Linseed cake was found to have a very low biological value. A supplementary

effect of the maintenance on the production ration was noted when hay was substituted for straw in the maintenance ration together with a production protein intake of poor biological value. With an excess of protein in the ration most of the excess nitrogen was secreted in the urine, while both the milk yield and the biological value of the food protein fell considerably. The authors suggest that together with an adequate amount of lysine in the protein ingested, a certain mixture of amino-acids is essential before maximum milk-yield can be obtained. S.T.C.

## FOOD VALUE OF DAIRY PRODUCTS

**117. The Effect of Commercial Drying and Evaporation on the Nutritive Properties of Milk.** K. M. HENRY, J. HOUSTON, S. K. KON, AND L. W. OSBORNE, Nat. Inst. for Res. in Dairying, Univ. of Reading, Reading, England. J. Dairy Res., *10:* 272–293, 1939.

Spray-dried (Kestner process), roller-dried and evaporated milks were prepared commercially from one batch of raw milk. The biological value of the proteins (nitrogen), vitamin B, and the growth-promoting properties of the milks were measured by tests on rats. Vitamin A and carotene, riboflavin and vitamin C were measured by physical and chemical methods.

The proteins (nitrogen) of evaporated milk were found to be significantly less digestible (true digestibility (91)) than those of spray-dried milk (true digestibility (94)). The difference between either of those and roller-dried milk (true digestibility (93)) was not statistically significant. The vitamin B content of evaporated milk (9 I. U./100 ml. of reconstituted milk) was only half that of spray-dried (18 I. U./100 ml.) and of roller-dried (19 I. U./100 ml.).

Reconstituted spray-dried, roller-dried and evaporated milks were fed *ad libitum*, supplemented with mineral, as the sole diet to groups of littermate male rats. Based on gains in weight per 100 ml. milk intake the dried milks were found to be significantly superior to the evaporated milk.

Processing the milk caused no loss in vitamin A and carotene. Moreover, they were not decreased after a year's storage of the processed milks. There was no difference in the riboflavin content of the processed milks and the factor was not affected by 16 months' storage.

Spray-drying caused a 20 per cent loss, roller-drying and evaporation a 30 per cent loss in the original vitamin C content of the milk. S.T.C.

**118. Studies on Digestibility of Proteins in Vitro. VII. Liberation of Cystine on Tryptic Digestion of Casein with Observations on the Instability of Cystine Toward Alkali.** D. BREESE JONES AND CHARLES E. F. GERSDORFF, Protein and Nutrition Res. Div., Bureau of Chemistry and Soils, U. S. Dept. of Agr., Washington, D. C. J. Biol. Chem., *129:* 207, 1939.

This paper is a presentation of data on the liberation of cystine from casein at various reactions and with tryptic digestion.

Cystine is readily liberated from casein by tryptic digestion at pH 8–9. A large part of the casein cystine, however, is destroyed during the digestion by the alkalinity of the digest. When the digestions are conducted at slightly acid reactions, cystine was quite readily and completely liberated, although at a slower rate than when the digests were alkaline. There was no destruction of cystine. About 40 per cent of the casein cystine was liberated in 24 hours at pH 6.6–6.8. At pH 6.8 nearly all the cystine was liberated in 120 hours, while 168 and 216 hours were required when the digestions were conducted at pH 6.6 and 6.2, respectively. K.G.W.

119. **The Effect of Orange Juice on Calcium Assimilation.** CAROLINE S. LANFORD, Dept. of Chemistry, Columbia Univ., New York City. J. Biol. Chem., *130:* 87, 1939.

Young growing rats were fed moderate quantities of orange juice in addition to a basal wheat and milk mixture. The amount of juice given daily (5 cc.) corresponded on the average to about 15 cc. per 100 Kilo calories of diet consumed.

The proportion of the calcium of the diet which was stored in the body under these conditions was about 8 per cent greater than the proportion stored by control animals on the basal diet alone. This improved assimilation of the dietary calcium was noted in every instance and was of unquestionable significance by the usual statistical criteria. K.G.W.

## ICE CREAM

120. **Formulas for Ice Cream with Reduced Carbohydrate Content.** W. J. CORBETT AND P. H. TRACY. Ill. Agr. Exp. Sta. Cir. 498, Nov., 1939.

Commercial ice cream ordinarily has a carbohydrate content of about 20 per cent and is unsuited for diabetic patients.

Three formulas for ice cream mix with low carbohydrate content are suggested. Directions are given for making the low-lactose milk which is used in two of the formulas.

Caloric values per pint of these ice creams drawn at 80 per cent overrun are shown.

A formula for home use is included. O.R.O.

121. **Plant Layout.** H. J. BROWN, Goldenrod Ice Cream Co., Chicago, Ill. Ice Cream Field, *34:* 4, 21, 26, Oct., 1939.

The author points out the advisability of planning the plant layout so that each operation can follow in proper routine with the elimination of

unnecessary handling of the products. He favors the one floor plan for manufacturing operations, and claims that continuous ice cream freezers are more suited for economical operation than batch freezers.

In general he states that all ice cream plants are similar, but that the peculiarities of each must be considered in order to be assured of efficient production. W.C.C.

122. **Raw Materials for Ice Cream.** J. Hoffman Erb, Dairy Dept., Ohio State Univ. Ice Cream Field, *34:* 4, 22, 23, Oct., 1939.

The author discusses the necessity of controlling the quality of raw materials used in the manufacture of ice cream. He points out that oxidized flavor is one of the most common defects in ice cream and states that from a practical point of view the elimination of copper from the mix is the most successful means of preventing it.

According to the author many plants now make Eh determinations on cream and in one instance cream is never frozen or stored for subsequent use if it has an Eh of 0.350. W.C.C.

123. **New Processes of Mix Manufacture.** E. C. Scott, Research Laboratory, Swift and Company, Chicago. Ice Cream Field, *34:* 4, 24–25, Oct., 1939.

The production of high quality ice cream at the lowest cost is the aim of the ice cream industry the author points out. It is stated that it is possible to produce high quality ice cream using either high or low percentages of serum solids, provided the proper balance is maintained between serum solids, stabilizers and other solids that will permit the ice cream to be drawn from the freezer in a stiff, fairly dry condition. When sweet cream is not available the tendency is towards the use of frozen cream in place of butter, although there is some indication that the findings of Leighton and Levitan (Ind. Eng. Chem., June, 1939) may tend to increase the use of high quality unsalted butter by first homogenizing the butter and skim milk to form cream.

It is claimed that soft ice cream as drawn from the freezer can often be traced directly to the use of stabilizers with a high sodium salt content.

The tendency to use higher sugar contents in ice cream has necessitated higher pasteurization temperatures. It is claimed that prolonged heating at temperatures from 150° F. to 180° F. results in the precipitation of calcium salts and this in turn impairs the body of the finished ice cream. The author states that pasteurizing and holding vats with stuffing boxes and horizontal type coils are becoming obsolete, due to the increased emphasis on sanitation.

It is stated that making ice cream mixes in the vacuum pan is not gaining in popularity. In the case of mixes made from mediocre quality prod-

ucts, it may have a slight advantage whereas otherwise it tends to make a flat-tasting mix.

Direct expansion cabinet coolers are rapidly replacing brine and sweet water coolers and immediate cooling to 34° F. to 40° F. is the general practice. Homogenizers of current design are either of the two stage type or else a single stage type with auxiliary features to give results that approximate double stage homogenization. The newer homogenizers generally accomplish satisfactory homogenization at lower pressures than was formerly required. According to the author the presence of fat globules larger than 3½ microns in diameter indicates incomplete homogenization. W.C.C.

124. **Modern Freezing Methods.** A. W. FARRALL, Creamery Package Manufacturing Co. Ice Cream Field, *34:* 4, 27, 28, 29, 44, Oct., 1939.

It is pointed out that the three principal requirements of freezing ice cream are:

1. The ice cream mix must be cooled rapidly to a temperature of from 22° F. to 24° F.

2. The proper amount of air must be incorporated uniformly in a fine state of division.

3. The freezer or its attachments must be capable of uniformly mixing fruits, nuts, candy and the like in the ice cream.

The author states that over-neutralization of the mix or the use of ingredients which contain a high percentage of sodium caseinate cause the ice cream to become sticky, thus increasing the power consumption of the freezer. Low whipping mixes cause difficulty in obtaining overrun with batch freezers and with continuous freezers may cause air slugs in the ice cream. Ordinarily as the temperature is lowered from 26° F. to 24° F. the ability to incorporate air is increased, whereas lowering the temperature below 23° F. air is whipped in with more difficulty and usually at 22° F. it is necessary to maintain a fairly high air pressure in order to obtain the desired overrun. This condition is made possible in the continuous freezers on the market.

The advantages of rapid freezing as a means of improving ice cream texture are mentioned and instructions for sharpening freezer blades properly are given and illustrated diagrammatically.

In modern freezing both batch and continuous freezers are in use. Efficient plant layout and arrangement of schedules contribute to economical plant operation. W.C.C.

125. **Hardening of Ice Cream.** P. H. TRACY, Dept. of Dairy Husbandry, Univ. of Ill., Ice Cream Field, *34:* 4, 30, 31, 44, Oct., 1939.

It is claimed that from 10 to 12 inches of good insulation is advisable in the walls and ceiling, and at least 8 inches on the floors of hardening rooms.

Ordinarily the hardening room should be large enough to hold about three times the amount of the peak day's run (roughly one per cent of the annual production). In the case of novelties and package goods, a larger space per gallon is necessary.

Three types of hardening rooms are mentioned: 1. Hardening rooms with shelf and ceiling coil arrangement, 2. Hardening rooms with bunker system, 3. Hardening rooms with the blower type of refrigeration unit. The merits of each type are mentioned.

The advantages of fast hardening are indicated and it is pointed out that air circulation in the hardening room markedly contributes to this. A brief review is given of a paper by Tracy and McGown (J. DAIRY SC. *17:* 47–60). W.C.C.

**126. Shrinkage of Ice Cream.** W. C. COLE, Dairy Industry Div., Univ. of Calif. Ice Cream Field, *34:* 4, 32–34, Oct., 1939.

The influence of changes in external pressure is considered by the author as contributing to the problem of ice cream shrinkage.

Products manufactured at sea level and shipped to altitudes five or six thousand feet above may increase considerably in volume, whereas those manufactured in higher altitudes and shipped to sea level often show shrinkage.

Experiments were carried out by placing samples of ice cream in a vacuum pan and observing changes in volume of the ice cream accompanying changes in pressure. The results are illustrated in tabular form and by means of photographs.

The author concludes as a result of this study that:

1. Variations in external pressure may cause marked changes in the volume of ice cream.

2. When such pressures are reduced, the volume of ice cream increases. When the pressure is increased again to the original value, ordinarily the ice cream does not return to its original volume; hence we say that it has shrunk. This shrinkage is the result of loss of air.

3. Some of the shrinkage in commercial ice cream particularly where marked changes in altitude are involved, may be explained on the basis of the principles just indicated.

4. When ice cream is subjected to these changes in pressure its hardness, which depends primarily upon temperature, is an important factor determining the extent to which shrinkage occurs. Other things being equal, the harder the ice cream the less will be its change in volume as a result of variations in pressure. W.C.C.

**127. Flavoring Characteristics of Individual Cocoa Varieties.** P. S. LUCAS AND I. A. GOULD, Michigan State College. Ice Cream Field, *34:* 5, 28, 29, 34, Nov., 1939.

The authors claim that the individual flavoring characteristics of the liquor from particular types of cocoa beans are unknown because of the custom manufacturers have of blending or combining various beans before or during their processing. They point out further that commercial chocolates and cocoas in many cases are modified by the addition during milling of vanillin, essential oil of almonds, cinnamon, cassia, ginger, tolu balsam and other materials. They selected seven representative varieties of cocoa beans on the New York markets. The average quality, production and price of these varieties are given in the following table. In addition they are described briefly.

*Average quality. Production and price of cocoas*

| Variety | Quality Rating | Production | Price N. Y. Market May 4, 1939 (cents) |
|---|---|---|---|
| 1. F. F. Accra | Sixth | First | 4.5 |
| 2. Arriba | Third | Fifth | 8.0 |
| 3. Bahia | Fifth | Second | 4.5 |
| 4. Puerto Cabello | First | Sixth | 12.0–18.0 |
| 5. Caracas | Second | Seventh | 10.0 |
| 6. Sanchez | Seventh | Third | 4.1 |
| 7. Trinidad | Fourth | Fourth | 7.5 |

Ice creams of the same composition except for the flavor used were prepared as a means of arriving at public preference for chocolate flavors peculiar to particular varieties. The authors report that their tests indicate the public preference for chocolate varieties in ice cream is for the stronger varieties of beans having chocolate flavor of the bitter type. They state that the commercial practice of blending several varieties of cocoa beans to avoid seasonal changes in flavor and to provide color and other desirable effects is justified. W.C.C.

**128. Public Health Standards for 1940.** DAVID LEVOWITZ, New Jersey Dairy Laboratories. Ice Cream Field, *34:* 4, 45–46, Oct., 1939.

The purpose of the article is to indicate not only what changes are to be expected in ice cream control during the next year, but also how the ice cream manufacturers may prepare for them. The author considered the following to be important:

*Ingredients*

a. *Dairy ingredients*—Standards applying to fresh fluid products for sale as such are to be applied to these products when used in the manufacture of ice cream.

b. *Non-dairy ingredients*—They must be of approved type, pure, and stored in such a manner that they are not contaminated, and given such treatment as is necessary prior to their addition to the mix.

*Processing Equipment and Practice*

a. *Pasteurization*—Heating to 155° F. (or higher) for 30 minutes by the "long hold" process; or where approved, 180° F. (or higher) for 16 seconds by high temperature-short hold process.

b. *Homogenization*—May be performed by any approved emulsifying mechanism. Only equipment which can be easily taken apart for cleaning and sterilization should be approved.

c. *Cooling, freezing, and packing*—The mix must be cooled to below 50° F., immediately after the conclusion of its (pasteurization or) homogenization. Molds for the freezing of bars, etc., are to be constructed without lead on surfaces coming into contact with the food products.

d. *Packages*—Metal cans used for bulk shipments must be made without lead on surfaces coming into contact with the food product. Metal containers must be cleaned and sterilized prior to use. Paper containers must be made of stock of the quality approved for the manufacture of milk containers. Assembly for containers must be done in an approved manner.

*Finished Product's Sanitary Standards*

a. *Samples*—Ice cream must be sampled with aseptic precaution from a full plant-package container, to determine the condition of the ice cream as it left the manufacturing plant.

b. *Phosphatase test*—Indophenol in phosphatase tests beyond that obligatory through controls is definite evidence of inefficient pasteurization.

c. *Total count*—A.P.H.A. tryptone-dextrose dairy nutrient agar is to be employed for the total colony count which must be well below 100,000.

d. *Direct microscopic count and types*—The standards employed for other dairy products will apply also to an area's ice cream.

e. *Coliform group significance*—Presence of this group indicates contamination. Confirmatory and corroboratory tests should be run to avoid the reporting of false positives.

The author discusses each of the factors listed above and points out further that considerable attention is now being given to ice cream dispensing sanitation. Those anticipating the installation of various types of equipment should anticipate future sanitation requirements. W.C.C.

**129. Let's Face Facts on Quick Frozen Foods.** C. Q. SHERMAN, C. Q. Sherman Corp. Ice Cream Field, *34:* 6, 12, 13, 51, Dec., 1939.

The author claims that ice cream manufacturers should be interested in frozen foods mainly because frozen food sales are greatest when ice cream

sales decrease. He claims the best type of outlet for frozen foods is the grocer or meat dealer catering to the average housewife. To begin with, he considers that only staple articles should be sold such as peas, beans, spinach, asparagus, strawberries, raspberries, etc.

It is pointed out that frozen foods must be displayed to be sold. The author warns against financing the dealers but recommends carrying insurance against mechanical breakdowns resulting in defrosting and spoilage. W.C.C.

**130. Determining Sanitary Quality by Laboratory Tests.** F. E. NELSON, W. J. CAULFIELD AND W. H. MARTIN, Kansas Agr. Exp. Sta. Ice Cream Field, *34:* 6, 14, 15, 16, 28, Dec., 1939.

The following factors, it is stated, are commonly used to measure ice cream quality: (1) taste, (2) physical characteristics such as body and texture, (3) quantitative factors such as amount of overrun, butterfat and solids content, and (4) the sanitary quality of the product. The sanitary quality is much more difficult to evaluate and control than the other factors listed, according to the authors.

They consider the number and types of bacteria present in ice cream to be due to (1) bacteria which enter the product at any point from the udder of the cow to the mouth of the consumer, (2) opportunities for contaminating organisms to grow once they have gained entrance, and (3) the destruction of bacteria by such means as pasteurization.

The authors report results of laboratory tests on 315 ice cream samples in which the latter were subjected to standard plate count for bacteria, the direct microscopic count, the Escherichia-Aerobacter or Coliform bacteria test, and the phosphatase test. The results obtained are presented in tabular form and their importance discussed. It is stated that the outstanding lesson to be obtained from the results is that a definite reason can be found for almost all plate counts above 50,000 per ml. and for many of the counts above 25,000 per ml. by using only these four simple laboratory tests.

In conclusion the authors state that there is no substitute for continuous care and vigilance on the farm, in the plant, and in the retail establishment but that a well run laboratory can do much to prevent irregularities in sanitary quality. W.C.C.

## MILK

**131. The Phosphatase Test for Control of Efficiency of Pasteurization.** H. D. KAY, R. ASCHAFFENBURG AND F. K. NEAVE, Imperial Bureau of Dairy Science, Shinfield, near Reading, England. Technical Communication No. 1, Oct., 1939.

The article is a critical review of the phosphatase test, covering com-

pletely the development, modifications and application of the test to milk and various milk products. It summarizes the results obtained by various investigators pointing out the advantages and limitations of the test, the sensitivity, and the effect of various factors such as storage, preservatives, bacterial contamination, stage of lactation, and mastitis on the reliability of the test.

The conclusions are that the test for milk is very sensitive, accurate, and effective in controlling the efficiency of pasteurization by the "holder" process. The test in its appropriate modification is sufficiently sensitive to detect a drop in temperature of 1.0 to 1.5° F. if pasteurizing temperature is 143° F. for 30 minutes. If 145° F. for 30 minutes a drop of 1.5° F. can be detected.

A reduction of 10 minutes in holding time can be detected but not 5 minutes with certainty.

The presence of 0.2 to 0.25 per cent of raw milk in pasteurized product can be detected and in many instances even smaller amounts.

Regarding the application of the phosphatase test to the high temperature short time holding process, it is concluded that, "the claim that the phosphatase test remains sensitive under H.T.S.T. conditions is well founded, but further experimental evidence is required to settle a number of details."

It is further concluded that the accuracy of the test when applied to products other than milk is still disputed, and further investigation is required with reference to amount of sample to be used for testing, limiting color standards, etc.

It is confidently expected that further research will achieve tests for milk products which will be as accurate and reliable for controlling the pasteurizing process as that already achieved for milk.

The bulletin also has an appendix giving precautions and detailed instructions for performing the phosphatase test. Technique for the following test and modifications are given.

A. Kay-Graham Test A and Test B.
B. Neave's Modification.
C. Gilcreas and Davis Modification.
D. Scharer's Modification.
    1. Laboratory Test
    2. Field Test
E. Leahy's Modification.
F. Aschaffenburg and Neave's Modification.

The price of the bulletin is two shillings. L.H.B.

**132. Accuracy of Methods of Sampling Milk Deliveries at Milk Plants.** P. H. TRACY AND S. L. TUCKEY. Ill. Agr. Exp. Sta. Bull. 459, 47–84, 1939.

A study of the accuracy of the methods used to sample the milk delivered at each of four dairies.

From the data secured from these dairies the following conclusions are drawn:

1. Inaccurate tests may result from improper mixing of the milk when dumped into the weigh tanks.

2. To determine the accuracy of sampling from weigh tanks, samples from each tank without previous stirring should be checked against samples taken when the milk has been thoroughly stirred.

3. Tests on composite samples properly taken and kept will give an accurate measurement of the fat content of the milk.

4. Periodic sampling would not be satisfactory on a market where variations in daily tests are wide.

5. Variation in daily tests on milk from the same patron was sufficiently great to indicate mechanical manipulation of the fat content.

6. The tendency for plant composite samples to test less than laboratory composite samples is thought to be due to variations from the accepted practice in the care of the samples.

7. Composite samples need not be taken in aliquot portions to give results that will be sufficiently accurate for practical purposes. O.R.O.

**133. Development and Present Status of the Big Milk Bottle.** ANONYMOUS. Milk Plant Monthly, *28:* 9, 24, 1939.

The demand for lower priced milk brought the large container into the milk distribution field. The development of the present most favored two-quart or half-gallon bottle from the gallon jug is discussed, giving consumer and distributor opinions. The gallon container with a handle appealed to the consumer because of its convenience in carrying, but distributor opinion was that the consumer who had not used the one gallon container would prefer the two-quart bottle at the same price per unit of milk. The majority of the distributors questioned favored the two-quart bottle over the gallon container. G.M.T.

**134. The Relation of Nutrition of the Cow to Development of Oxidized Flavor in Milk.** J. L. HENDERSON, Div. Dairy Ind., Univ. of Calif., Davis, Calif. Milk Plant Monthly, *28:* 12, 26, 1939.

Eighteen papers on oxidized and rancid flavors in milk are reviewed showing that rations high in carotene result in a milk less susceptible to metal-induced oxidizer flavor and to the development of rancidity. For this purpose carotene concentrate, carrots and various green feeds have been used successfully. Maintaining a satisfactory carotene content in the milk throughout the year seems now to be the part of proper dairy herd managament, thus assuring not only a more stable milk but aids in maintaining a uniform vitamin A potency as well. G.M.T.

**135. A Comparison of the Imperviousness of Commonly Used Paper Milk Containers.** R. B. STOLTZ AND T. V. ARMSTRONG, Dept. of Dairy Techn., Ohio State Univ., Columbus, Ohio. Milk Plant Monthly, *28:* 12, 54, 1939.

Five different makes of quart paper bottles two of square and three of cone construction were studied for absorption of liquids and penetrability of dyes. None of the bottles studied was found to be impervious. Two of the three bottles paraffined in the factory, cone shaped, offered the best protection against the entrance of moisture to the fibre. There was a tendency toward greater absorption of moisture at the higher temperatures. A 40 per cent cream seemed less penetrable than homogenized milk or skim milk. The weight increase per bottle ranged from 0.11 grams to 1.55 grams.

G.M.T.

**136. Physico-Chemical Principles Involved in Controlling Cream Body.** H. H. SOMMER, Univ. of Wis., Madison, Wis. The Assoc. Bull., Intern. Assoc. Milk Dealers, 32nd year: 113–117, Dec., 1939.

Attention is drawn to the complex nature of milk and an extensive catalogue made of conditions promoting or decreasing viscosity. The structure of cream is conceived to be due to clusters of fat globules in contact, one with another. The difference in effect when the adsorbed film is acquired by solid as compared with liquid fat globules is emphasized. A hypothesis is given to explain the functioning of the cooling-warming-cooling method of increasing viscosity from a physico-chemical viewpoint.

E.F.G.

**137. Studies on the Structural Aspects of Paraffined Paper Containers.** C. S. MUDGE, D. C. FOORD, J. L. HENDERSON. The Assoc. Bull., Intern. Assoc. Milk Dealers, 32nd year: 107–112, Dec., 1939.

Completeness of paraffining of paper milk containers was measured by extent of staining of the board when an iodine solution was allowed to remain in the container for 5 minutes. Containers stored at various humidities for 2 to 3 weeks were examined bacteriologically, but no evidence was obtained that bacteria work to the surface of the paraffin layer. Sediment tests made on paraffin films dissolved off with gasoline showed negligible sediment. Unbleached paper stocks resulted in higher ascorbic acid values and less impairment in flavor when the milk was exposed to direct sunlight for two hours than when bleached paper was used. An inner layer of heavily dyed paper is suggested as a possible improvement.

E.F.G.

**138. Microscopic Examination of Pasteurized Milk.** H. MACY, Univ. of Minn., St. Paul, Minn. The Assoc. Bull., Intern. Assoc. Milk Dealers, 32nd year: No. 4: 97–106, Dec., 1939.

The standard plate method and the Breed microscopic method were used on "street" and "plant" samples of pasteurized milk and on milk pasteurized in the laboratory. Dividing 275 "street" samples into six groups in the low count group the ratio of the plate to microscopic count was 1:260 while in the high count group the ratio was 1:25. The plant and individual patron samples followed somewhat the same general pattern. Laboratory pasteurized samples commonly decreased in microscopic count at least 50 per cent following pasteurization. Thermophyles were revealed by a microscopic count which were not fully accounted for by the regular plating method for the determination of thermophyles. The results suggest that microscopic examination of milk may yield some helpful information. The types of bacteria in the raw milk will have a good deal to do with the results obtained by microscopic examination of the pasteurized milk.

E.F.G.

## MISCELLANEOUS

**139. The Distributor Is the Chief Factor in the Successful and Adequate Consumption of Dairy Products.** MRS. W. E. FRIBLEY, Chicago Housewives League, Milk Plant Monthly, *28:* 6, 52, 1939.

Price alone does not seem to be the chief factor in the purchase and utilization of more milk by the average housewife, but rather indifference to dairy products. The consumer must be interested in dairy products, not by the milkman whom no housewife considers the proper person to educate her to use more dairy products, but through effective advertising, placards, pictures, radio programs, menus and luncheon parties. Many mothers know the children should receive a quart of milk a day but grow weary of trying to induce them to drink it, since the children themselves have not been interested. The suggestion is made for the need of adequate quick cooking dairy dishes, for more information concerning the uses of milk, and for protected doorstep delivery as aids in increasing consumption of dairy products in the home.

G.M.T.

**140. Looking into This Washing Powder Problem.** A. H. BAYER, Nat. Dairy Products Corp., Schenectady, N. Y. Nat. Butter and Cheese J., *30:* 11, 52, 1939.

For good cleaning and prolonging of life of equipment study each cleaning job, do not use washing powder in excess, see that equipment is properly washed, be sure equipment is left dry after cleaning and sterilizing, get a reliable vendor to survey your requirements and use his service.

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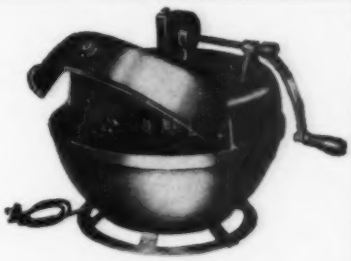

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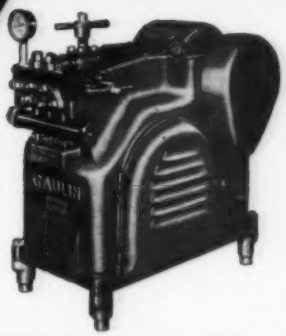

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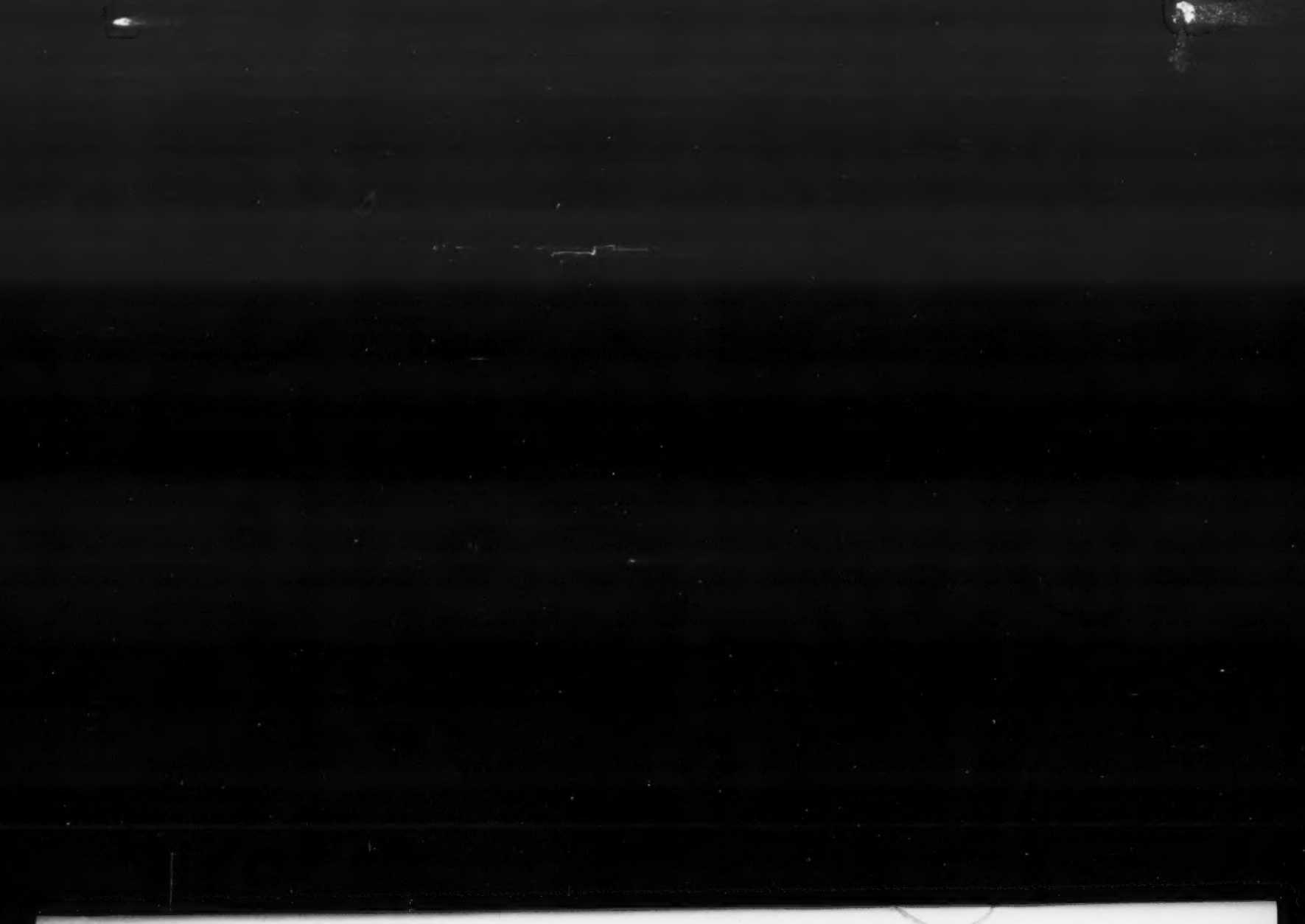

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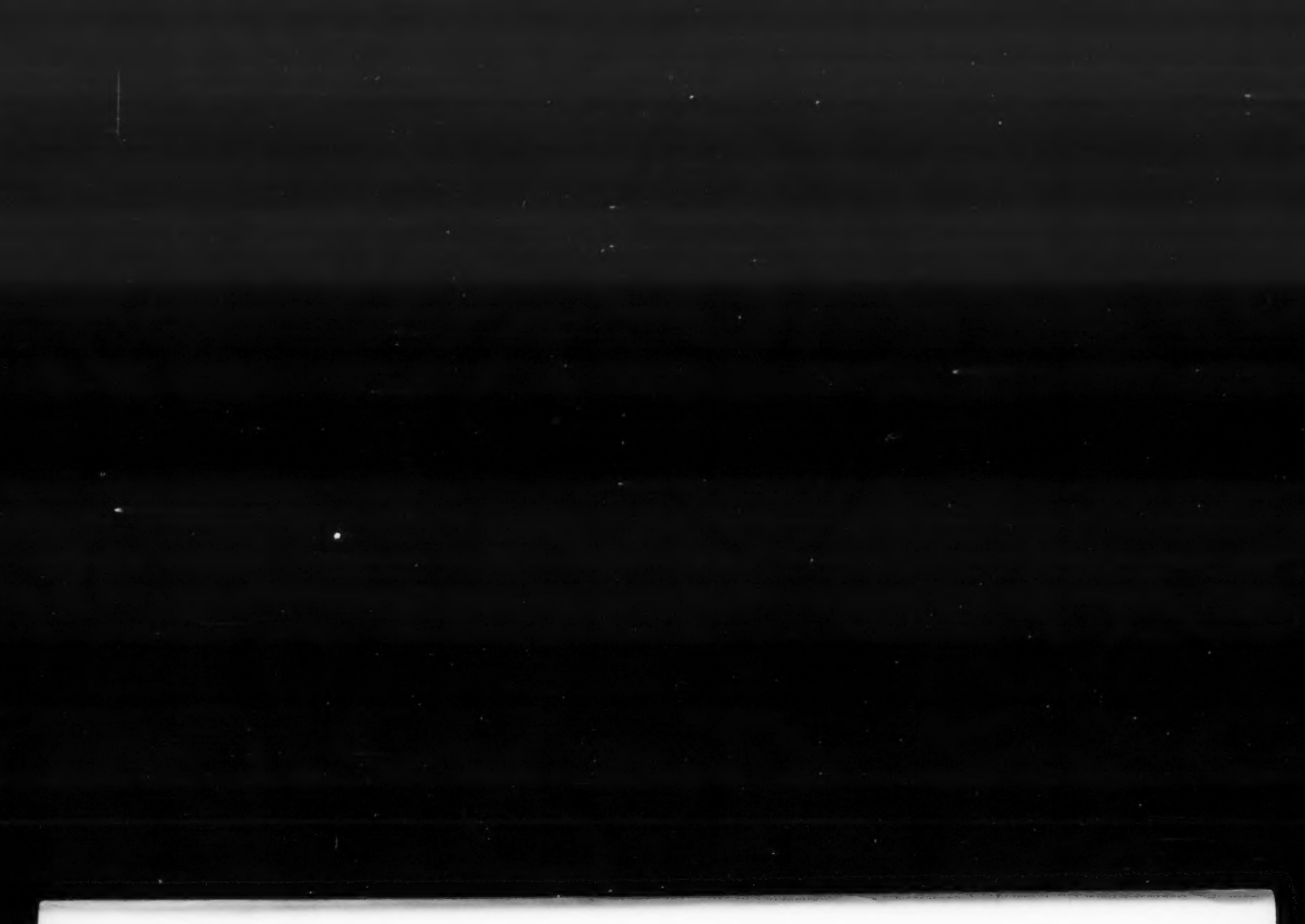
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